50th
Oilseed Conference

Tools for Improving Profits

January 21–23, 2001 ✸ Hyatt Regency New Orleans

Proceedings

50th Oilseed Conference

January 21–23, 2001 ✦ Hyatt Regency New Orleans ✦ New Orleans, Louisiana, USA

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Michael K. Dowd, SRRC/ARS/USDA

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Session Chairpersons:
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Lynn A. Jones, National Cottonseed Products Association, Inc.
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Program Schedule

IMPORTANT NOTICE

The papers in these proceedings have been reproduced exactly as submitted by the authors.

PROGRAM SCHEDULE

Sunday, January 21, 2001

3:00 p.m.	Registration Opens
5:30 p.m.	Welcome Reception
5:30 p.m.	Visit with table top exhibitors and poster authors

Monday, January 22, 2001

7:30 a.m.–5:00 p.m.	Registration
7:30 a.m.–8:30 a.m.	Continental Breakfast
8:30 a.m.	Opening Remarks

INVOCATION

David H. Kinard
National Cottonseed Products Association, Memphis, TN

CALL TO ORDER BY GENERAL CHAIRPERSON

Robert L. Stroup
R.L. Stroup Co. Ltd., Troy, OH

KEYNOTE PRESENTATION:

Biotechnology from the Food Industry Perspective
Susan K. Harlander, BIOrational Consultants Inc., New Brighton, MN

SESSION I
Oilseeds Roundtable
Outlook and Comments from the Commodities

Monday Morning—January 22, 2001

Session Chairperson:
Lynn Jones, The National Cottonseed Products Association, Inc., Memphis, TN

9:30 a.m.	**A.**	**United Soybean Board.** John Becherer, United Soybean Board, St. Louis, MO
9:45 a.m.	**B.**	**Canola Council of Canada.** Dale Adolphe, Canola Council of Canada,
10:00 a.m.	**C.**	**National Sunflower Association.** Larry Kleingartner, National Sunflower Association, Bismark, ND
10:15 a.m.	**D.**	**National Cottonseed Products Association.** Lynn Jones, National Cottonseed Products Association, Memphis, TN
10:30 a.m.		*Coffee Break*
11:00 a.m.		**Roundtable Discussions with Panelists**
11:45 a.m.	**E.**	**MACT Regulation Update.** Ron Moeller, Cargill, Minneapolis, MN

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LUNCHEON
Monday, January 22, 2001
12:15 p.m.

Dedicated time to view poster presentations and visit with table top exhibitors.

SESSION II

Researching and Advancing New Technologies

Monday Afternoon—January 22, 2001

Session Chairperson:
John P. Cherry, Eastern Regional Research Center, ARS, USDA, Wyndmoor, PA

2:00 p.m.	**F.**	**Future of Drought Resistant Oilseed (Soybean) Cultivars.** Thomas E. Carter, USDA, North Carolina State University, Raleigh, NC
2:30 p.m.	**G.**	**Extrusion-Expelling and Minimal Oil Refining for Processing Identity-Preserved Soybeans.** Tong Wang, Iowa State University, Ames, IA
3:00 p.m.	**H.**	**Soapstock Utilization: A Process for the Production of Biodiesel Fuel.** Michael J. Haas, Eastern Regional Research Center, USDA, ARS, Wyndmoor, PA
3: 30 p.m.		*Coffee Break*
4:00 p.m.	**I.**	**Value-Added Adsorbents from Oilseed Hulls.** Wayne E. Marshall, Southern Regional Research Center, USDA, ARS, New Orleans, LA
4:30 p.m.	**J.**	**Effects of Oilseed (Peanuts) on Appetite, Food Intake, Energy Balance and Cardiovascular Risk.** Richard Mattes, Purdue University, West Lafayette, IN

50TH OILSEED CONFERENCE
Day Two
Tuesday Morning—January 23, 2001

7:30 a.m.	Registration Opens
7:30 a.m.–8:30 a.m.	Continental Breakfast

SESSION III
Management
Tuesday—January 23, 2001

Session Chairperson:
Grant Mitchell, Process Plus, Cincinnati, OH

8:30 a.m.	**K.**	**Project Specific Web Sites.** Grant Mitchell, Process Plus, Cincinnati, OH
		Internet Tools for Improving Profits. R.L. Stroup Company Ltd., Troy, OH
9:00 a.m.	**L.**	**Hiring and Retaining Good Employees.** Patricia Bivins, Equal Opportunity Commission, New Orleans, LA
9:30 a.m.	**M.**	**Partnerships to Reduce Pollution from Agricultural Activities.** Carlton Dufrechou, The Lake Pontcharatrain Basin Foundation, Metairie, LA
10:00 a.m.		*Coffee Break*
10:30 a.m.	**N.**	**Incident Database and Macroanalysis to Help Safety Direction.** John A. McIntosh, III, Procter & Gamble, Cincinnati, OH
11:00 a.m.	**O.**	**Process Safety Management and Hidden Benefits.** Ron Collier, Process Plus, Cincinnati, OH

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LUNCHEON
Tuesday, January 23, 2001
11:30 a.m.

GRAND PRIZE DRAWING AT LUNCHEON

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Poster Session

POSTER SESSION

Sunday, January 21, 2001—5:00 p.m.–7:00 p.m.
Monday, January 22, 2001—12:30 p.m.–1:30 p.m.

Session Chairperson: Michael K. Dowd, USDA, ARS, Southern Regional Research Center, New Orleans, LA

Posters will be on display throughout the conference.
Be sure to visit with the poster presenters at the times noted above.

P. **Modification of Vegetable Oils Using Tung Seed Extracts.** John M. Dyer, Dorselyn C. Chapital, Jui-Chang W. Kuan, and Armand Pepperman, USDA, ARS, Southern Regional Research Center, New Orleans, LA

Q. **Oilseed and Grain Processing at the Southern Regional Research Center.** Robert J. Hron, Sr. and Michael K. Dowd, Southern Regional Research Center, New Orleans, LA

R. **Antioxidants and Sunscreen Products from *Veronia galamensis* Epoxidized Triacylglyceride.** Reuben R.K. Kimwomi[1], Gerhard Kossmehl[1], Eldar B. Zeinalov[2], P.M. Gitu[3], and B.P. Bhatt[3], [1]Institut of Organic Chemistry of the Free University, Berlin, Germany; [2]Academy of Science of Azerbaijan, Baku, Azerbaijan; and [3]University of Nairobi, Nairobi, Kenya

S. **Synthesis and Physical Properties of Some Symmetrical Disaturated Monounsaturated Triacylglycerols and Their Functional Roles in Food Oil Systems.** G.R. List, K.R. Steidley, and R.O. Adlof, National Center for Agricultural Utilization Research, Peoria, IL

T. **A Comparison of Corn Oils: Corn Germ Oil, Corn Kernel Oil, and Corn Fiber Oil.** Robert A. Moreau[1], Vijay Singh[2], Robert J. Hron Sr.[3], and Kevin B. Hicks[1], [1]Eastern Regional Research Center, ARS, USDA, Wyndmoor, PA; [2]University of Illinois, Champaign, IL; and [3]Southern Regional Research Center, New Orleans, LA

U. **Expression of Three Omega-3 Fatty Acid Desaturase Genes from Tung Seeds.** Hurley Sheperd[1], John Dyer[1], Fuqiang Tang[2], Ding Shih[2], and Armand Pepperman[1], [1]USDA, ARS Southern Regional Research Center, New Orleans, LA; and [2]Louisiana State University, Baton Rouge, LA

V. **Bench Scale Production of Monolaurin from Coconut C_{12} Fatty Acid.** Carmelita H. Viernes, Industrial Technology Development Institute, Manila, Philippines

W. **Effect of Acid Treatment of Fuzzy Cottonseed on Free Fatty Acid Content Determination.** P.J. Wan[1] and Don Britton[2], [1]Southern Regional Research Center, ARS, USDA, New Orleans, LA; and [2]Mid Continent Labs, Memphis, TN

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Table Top Exhibits

PARTICIPATING EXHIBITORS

Alfa Laval, 200 South Park Blvd., Greenwood, IN 46143. Alfa Laval is a supplier of process equipment for the edible oil industry. Products include high speed separators, decanters, mixers and Soft Column deodorizers. Alfa Laval has recently introduced a new high capacity PX-separator. Separators are available for all edible oil processes. Alfa Laval supplies complete processes, including miscella refining, degumming, neutralization, dewaxing and deodorization.

Bratney Companies, 3400 109th Street, Des Moines, IA 50322. At Bratney Companies, we provide turnkey engineering, design and construction services, and equipment solutions, for the finest seed, feed, grain, oilseed, and milling facilities world-wide. We maintain our position in the industry through integrity, creativity, and reliability.

Buhler Inc., 1100 Xenium Lane, Plymouth, MN 55441. Representatives will be on hand to discuss Buhler's complete range of oilseed processing equipment and turnkey plant capabilities. Buhler manufactures preparation, meal grinding, cleaning, unloading and storage equipment.

Campro International Inc., 1113 Grandeur Crescent, Oakville, Ontario L6H 4B4 Canada. Since 1972, Campro International has been supplying the oil industry with leading technologies in chemical and physical refining. Our specialty is compact, pre-assembled plants, 5-50 tons/day, as container-shipped modules for on-site re-connection, requiring only utilities, a standard factory-type floor, and a building envelope, to be operational.

Crown Iron Works Company, P.O. Box 1364, Minneapolis, MN 55440. The Crown Group provides complete design and supply services for oilseed and edible oil processing. Crown's Oilseed Division specializes in solvent extraction, refining, methyl ester and oleochemical technology. Crown has offices in England, Brazil, Honduras, Moscow and China to service their worldwide customer base.

The Essmueller Company, P.O. Box 1966, Laurel, MS 39441. Since 1978, The Essmueller Company manufacture drag conveyors, bucket elevators and turnhead distributors for the industrial processing industries. Now introducing our new and innovative Model ET and Patent Pending Model EM enclosed belt conveyors with capacities exceeding one million bushels per hour.

Hutchison-Hayes Separators, Inc., 3520 East Belt, Houston, TX 77015. Hutchison-Hayes Separator specializes in spare parts, service, and repairs for the vertical high speed separators. Hutchison-Hayes also manufactures decanting centrifuges and reconditions used centrifuges. Hutchison-Hayes has also developed and made patent applications for a process system to reclaim waste fluids by using Ultra-Filtration.

Industrial Filter & Pump Mfg. Co., 5900 W. Ogden Avenue, Cicero, IL 60804. Filtration equipment for curde and refining oil processing featuring our type 122 horizontal tank, vertical leaf filters, type 112 vertical tank, vertical leaf filters, and our type 199 filter presses.

Krupp Elastomertechnik GmbH, Division Edible Oil Technology, Seevestr. 1, 21079 Hamburg, Germany. Krupp Elastomertechnik, Division Edible Oil Technology covers the whole range of components for processing oilseed and crude oil, from seed preparation and pressing to solvent extraction and oil refining with degumming, bleaching, winterization and deodorization. Most famous Krupp products are the EP-Series Screw Presses and the Carrousel Extractor.

Laidig Industrial Systems, 14535 Dragoon Trail, Mishawaka, IN 46544. Laidig manufactures bottom unloading equipment for both cone and flat bottom silos and bins from 6' to 60' diameter. Our unloaders may be installed in new silos designed for our equipment or retrofitted into existing structures. They are designed to unload difficult to handle materials even if the silo is full.

LFC North America, 20000 Governors Drive, Olympia Fields, IL 60461. As an innovative engineering and manufacturing company, LFC has developed and perfected many filtration products. Specialized experience allows LFC to customize the filter configuration, whether horizontal or vertical pressure leaf, pulse tube, or one of our other quality products to suit the needs of our customers. As the most critical part of the filter, LFC gives special attention during the manufacturing of the filter elements which can be produced for all makes of vessels.

Lurgi PSI Inc., 1790 Kirby Parkway, Suite 300, Memphis, TN 33138. Lurgi representatives will present their experience in design, engineering, and construction of state-of-the-art processing units for oilseed extraction, oil refining and modification and for oleochemicals. With its global organization, Lurgi has serviced its customers around the world for more than 100 years.

Riley Equipment, Inc., 2205 South Old Decker Road/PO Box 435, Vincennes, IN 47591. Riley Equipment, Inc. is the manufacturer of dry bulk handling equipment.

Tramco Inc., 1020 E. 19th Street, Wichita, KS 67214. . For over 30 years, TRAMCO has been involved in the design, application, engineering, and manufacturing of the most complete line of chain conveyors, enclosed belt conveyors, and specially designed conveyor and conveyor conversions. Tramco begins with sound engineering design, producing conveyors of exceptional quality and durability.

Trinity Consultants, 12801 N. Central Expressway, Suite 1200, Dallas, TX 75243. Trinity Consultants is a nationwide firm that assists industrial facilities with air quality, industrial risk management, and environmental information management services. Our services to the oilseed processing industry include: New Source Review air permitting, Clean Air Act compliance, atmospheric dispersion modeling, NPDES Clean Water Act permitting, and Storm Water Pollution Prevention Plans.

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Door Prizes

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Door Prize Package	Donated By	Value	Raffle Ticket
Browning BPS 12 Gauge Shotgun	50th Oilseed Conference Organizing Committee	$700.00	Orange
Garmin eTrex GPS Personal Navigator & Leatherman Micra Tool	Buhler Inc.	$150.00	Blue
Porter-Cable 12V Cordless Drill/Driver with flashlight, 2 Batteries, Charger & Case	Campro International	$150.00	Blue
Bushnell 7 x 50 Binoculars & Storm Proof Electronic Lighter	Crown Iron Works Company	$150.00	Blue
Tournament Leader Fish Finder & Storm Proof Electronic Lighter	The Essmueller Company	$150.00	Blue
SKB Golf Bag Travel Case, Groove Doctor Brush, and 2 College Golf Balls	Laidig Industrial Systems	$150.00	Blue
2 Motorola Talkabout 2-Way Radios (Model 250)	LFC North America	$150.00	Blue
Palm M100 Handheld PDA	Lurgi PSI Inc.	$150.00	Blue
Bazuka Adjustable Rod Case, Shimano Compre Graphite Rod (6.5'), Shimano Symetre 2000 Reel, Stren Line (6# test, 100 yds.), and Nor-Mark K-Steel Fillet Glove	Trinity Consultants	$200.00	Blue

How to Win A Door Prize:

1. You received 1 orange and 1 blue raffle ticket at registration.
2. The orange ticket is for the Shotgun and the blue ticket is for your choice of one of the other door prize packages.
3. On Tuesday morning, each of the companies will be showcasing their door prize at their tabletop exhibit.
4. Drop your blue ticket at the door prize package you want to win. Drop your orange ticket at the rifle display at the registration table.
5. The winners will be drawn at Tuesday's luncheon.
6. You must be present to win.

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United Soybean Board

John Becherer
United Soybean Board
St. Louis, MO

Commodity Roundtable: Soybeans

John Becherer

Chief Executive Officer

United Soybean Board (USB)

St. Louis, Mo.

www.unitedsoybean.org

Commodity Roundtable: Soybeans

Discussion Points

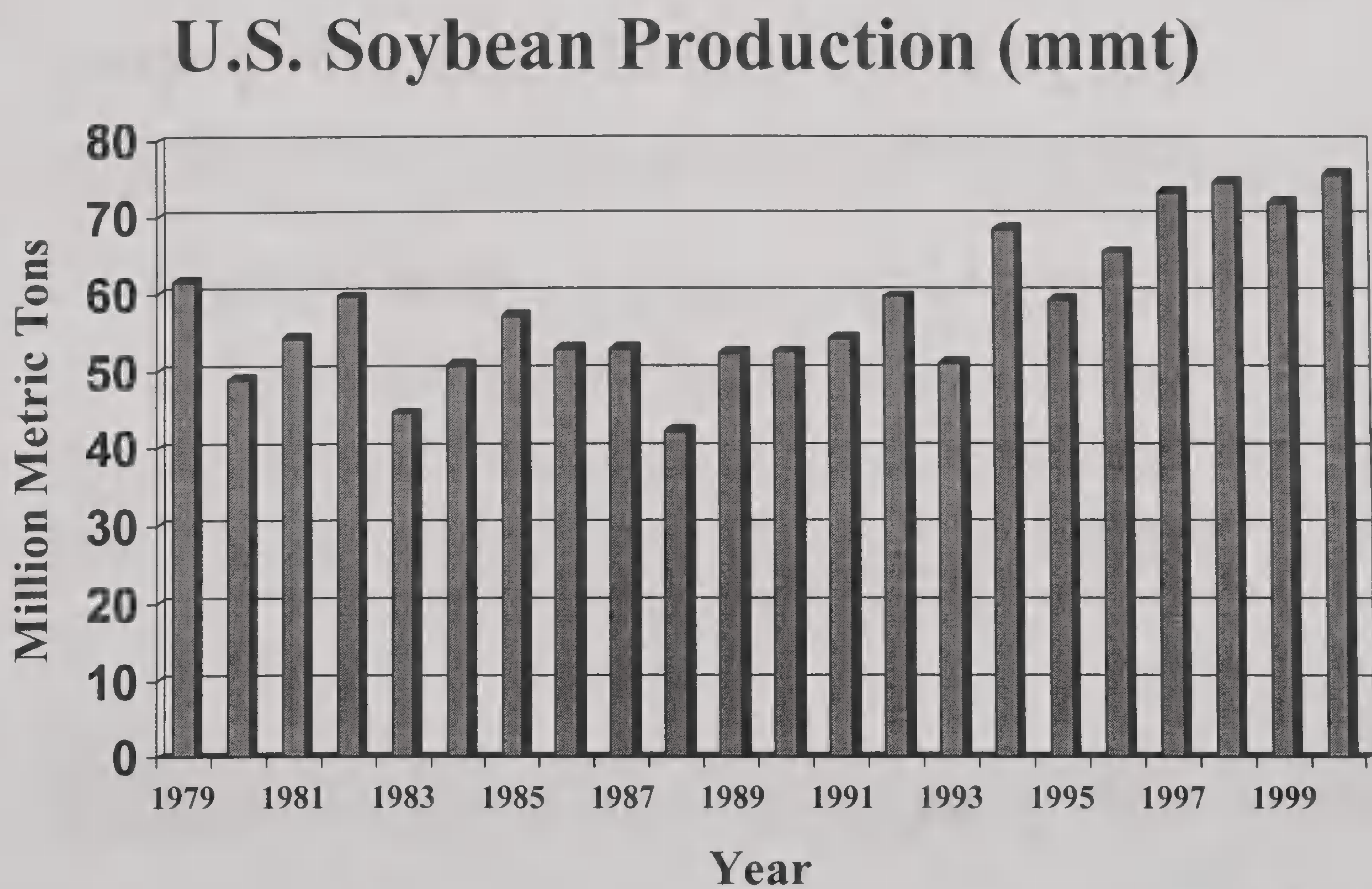
- Soybean Checkoff: Effective, Efficient, Farmer-driven
- Soybean Production in 2001
- Soybean Checkoff Efforts to Increase International & Domestic Utilization
- Soybean Checkoff's Better Bean Initiative
- Biotechnology

Commodity Roundtable: Soybeans

Soybean Checkoff

- Soybean farmers validate support
  - <3% of eligible farmers request referendum: USDA, May, 2000
- 0.5 percent of the market price per bushel sold deducted at point of sale
- 61 farmer-directors from 29 states and regions
- 10th Anniversary in 2001

Commodity Roundtable: Soybeans

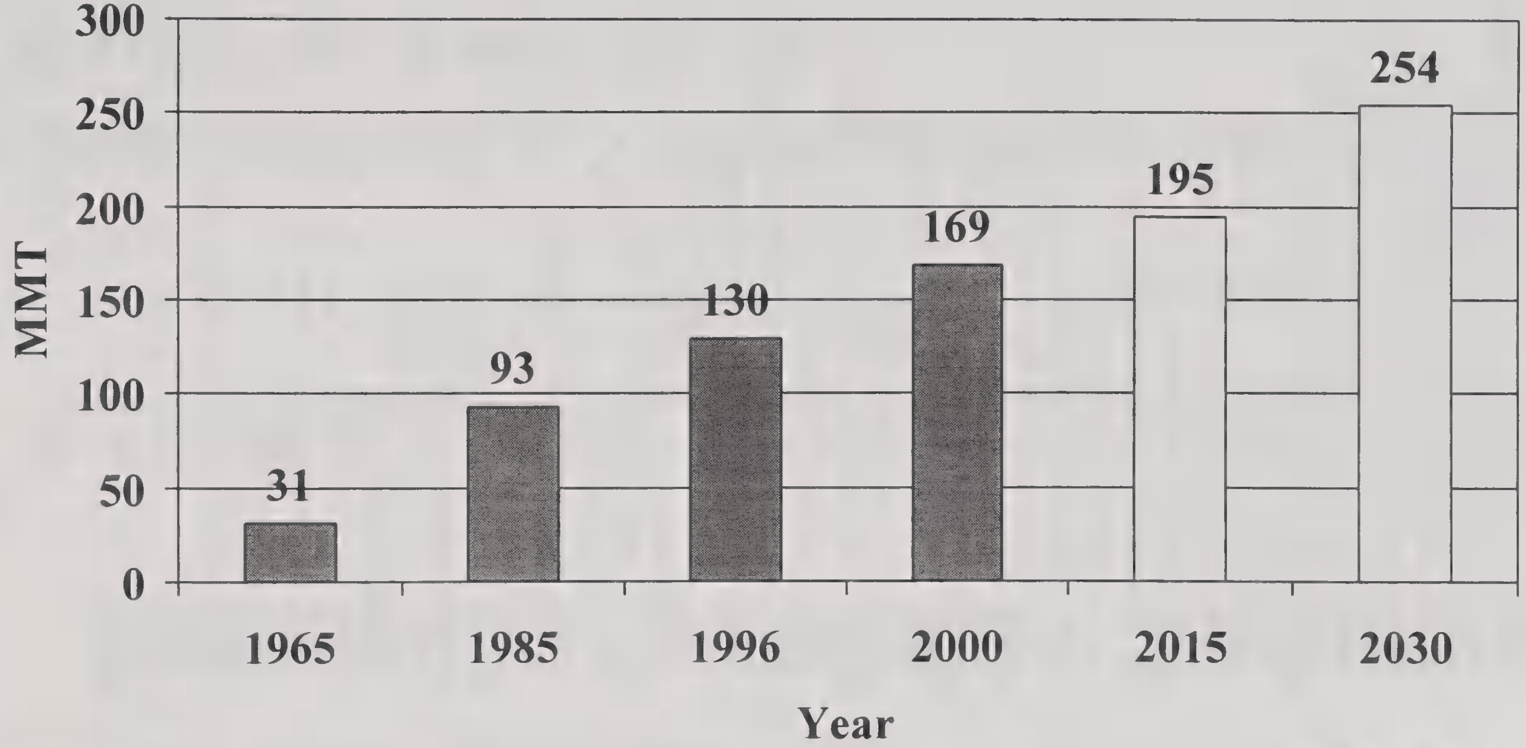


Source: John Baize & Associates

Commodity Roundtable: Soybeans

Global Soybean Production (mmt)

FAO Estimates for 2015 & 2030



Source: John Baize & Associates

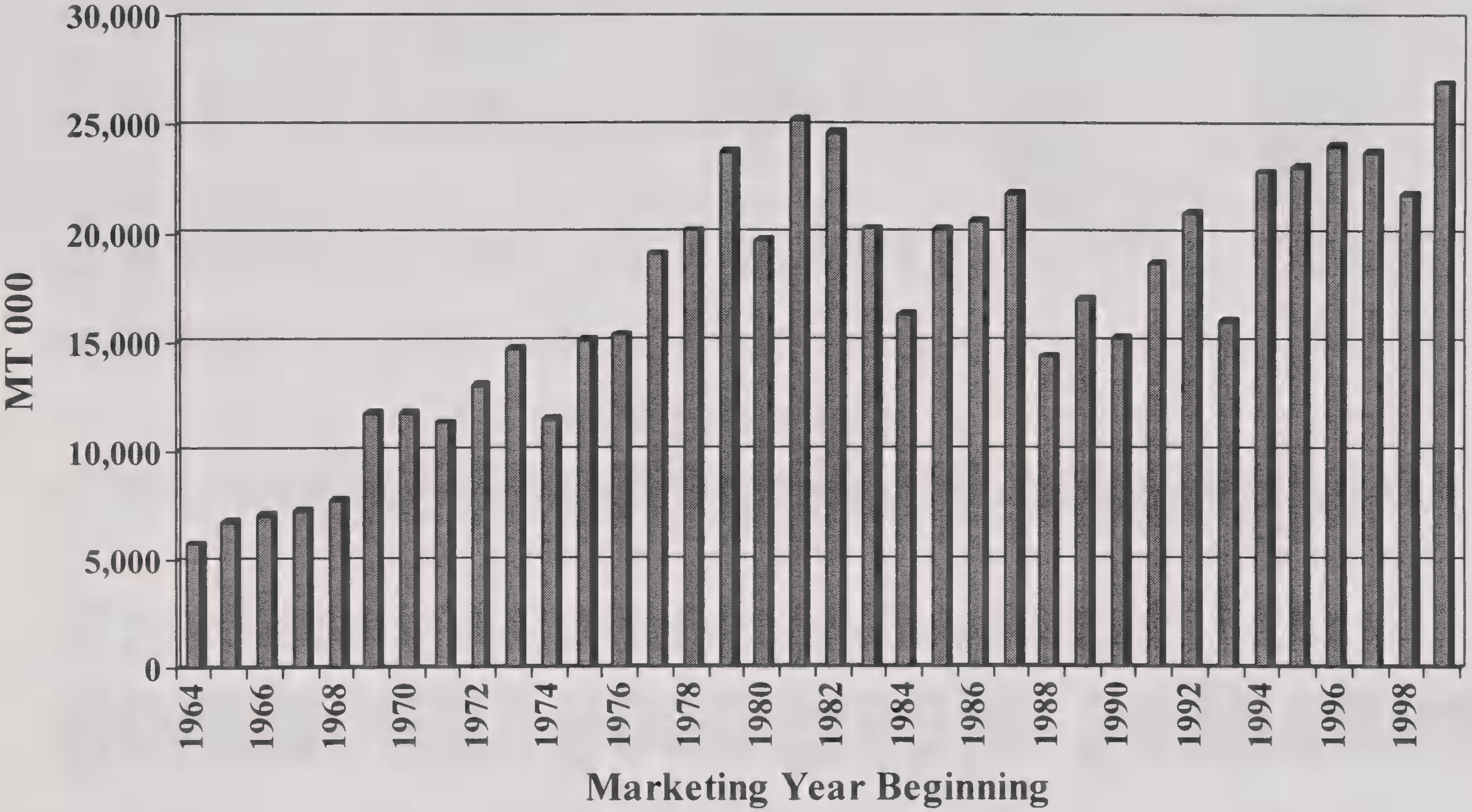
Commodity Roundtable: Soybeans

Soybean Checkoff: International Marketing

- Checkoff-funded activities in 80 countries
- 26.97 mmt of U.S. soybeans exported in Marketing Year '99/'00
  - all-time record
- $10 million invested by checkoff in FY2001
- Upward trend anticipated
  - China
  - Europe

Commodity Roundtable: Soybeans

U.S. Soybean Exports

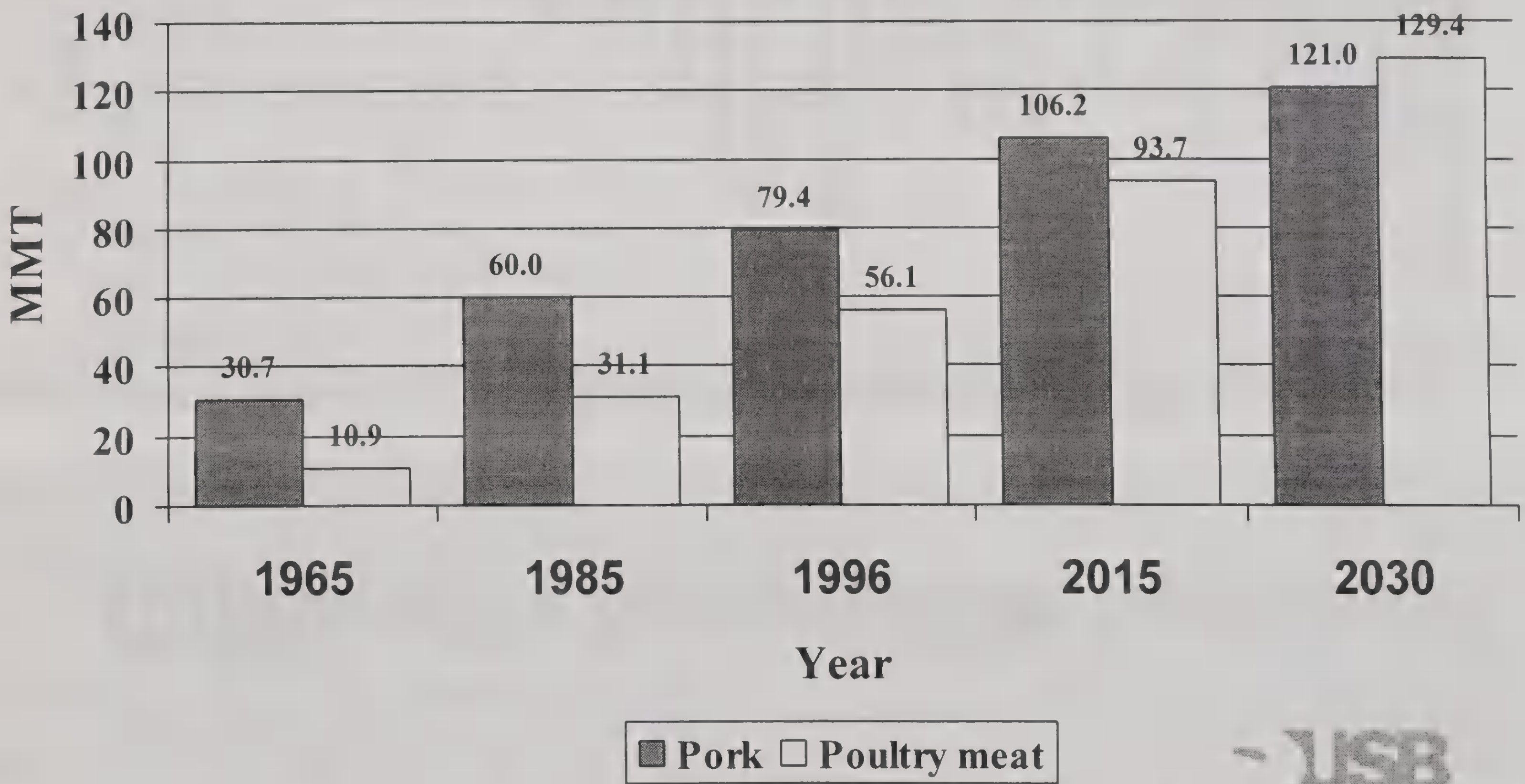


Source: John Baize & Associates

Commodity Roundtable: Soybeans

Global Pork & Poultry Production

FAO Estimates for 2015 & 2030



Commodity Roundtable: Soybeans

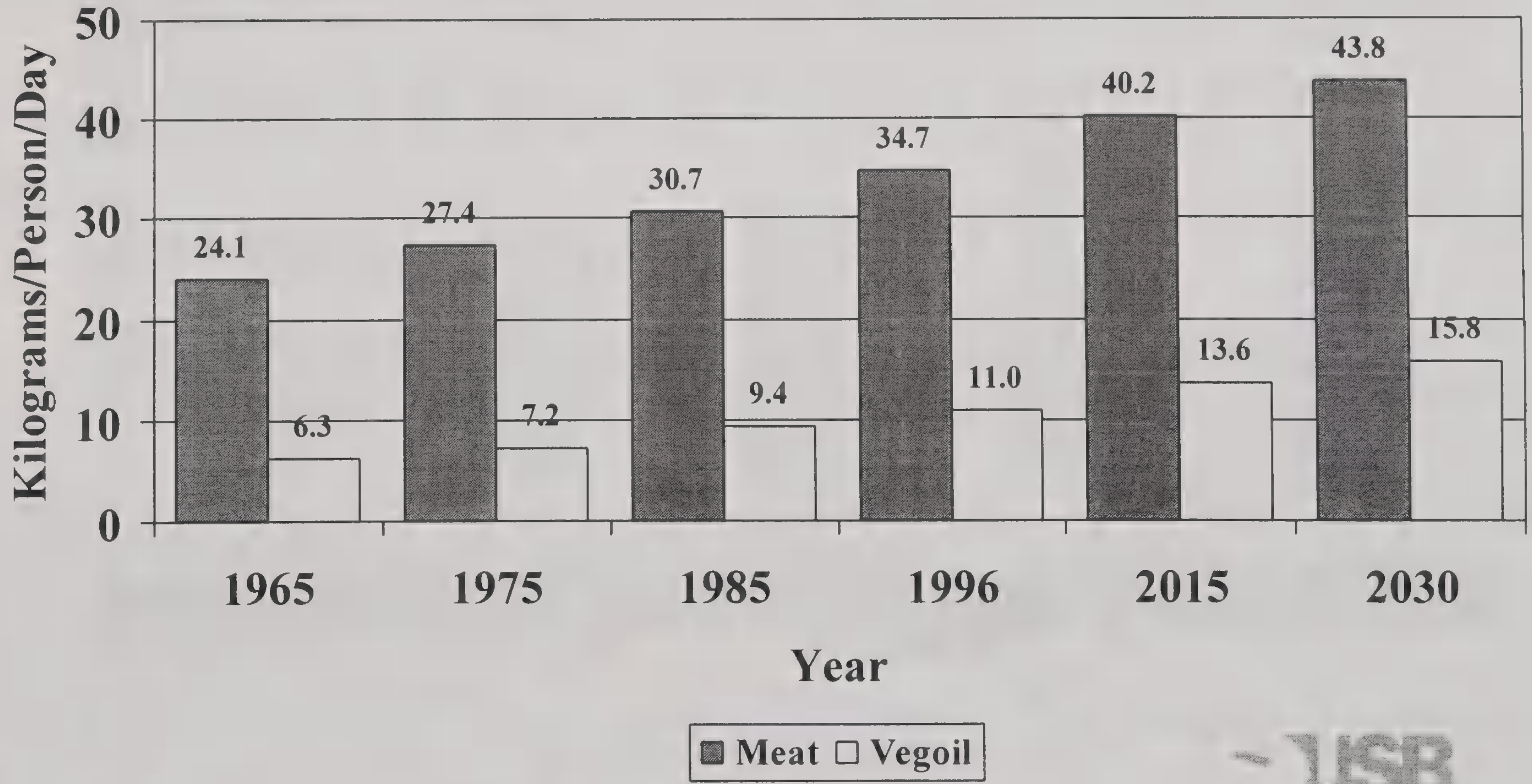
Soybean Checkoff: Domestic Marketing

- Exceeds 1.6 billion bushels
- Funding meat and poultry export efforts
- Promoting domestic oil utilization
- Promoting domestic soyfoods consumption
- Successful health research

Commodity Roundtable: Soybeans

Per Capita Meat & Vegoil Consumption

FAO Global Estimates for 2015 & 2030



Commodity Roundtable: Soybeans

Checkoff Consumer Attitudes Toward Soy Annual Survey

- 76% of consumers consider soyfoods to be healthy
- 27% of consumers eat soy products at least once per week
- 35% of consumers know about health claim
- Soybean oil gets high marks

USB

Commodity Roundtable: Soybeans

Checkoff Health Research Program

- Targets links between soy consumption and reduced risk of osteoporosis and prostate cancer
- $10,000 award for proposal helped secure $1.5 million from NIH for soy and osteoporosis prevention research
- FY '01 program also targets research on optimum consumption levels of soy isoflavones

UNITED SOYBEAN BOARD

Commodity Roundtable: Soybeans

Soybean Checkoff: New Uses

- Targets soy-based lubricants, plastics, coatings & inks, adhesives and solvents
- 15 new products or applications introduced in FY'00
- Goal is 8 new industrial uses by 2005 that consume at least 10 million bushels
- Federal bio-based initiative

Commodity Roundtable: Soybeans

Soybean Checkoff: Production Research
Better Bean Initiative

- Responding to changing needs of industry partners
- Stack improved traits to improve composition
- ISEO endorsement of ARS low-sat/low-lin variety
- ARS to plant in '01

Commodity Roundtable: Soybeans

<u>Soybean Checkoff: Biotechnology</u>

- **<u>All</u> commercially available biotech *soybean* varieties have been approved for feed and food use in U.S. <u>and</u> abroad**
- **Since '96, USB & ASA have urged seed industry to secure approvals <u>before</u> commercialization**
- **$3 million spent abroad to provide information**
- **Founding partner of ACC**

Commodity Roundtable: Soybeans

Thank You

United Soybean Board (USB)

St. Louis, Mo.

www.unitedsoybean.org

USB

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Canola Council of Canada

Dale Adolphe
Canola Council of Canada
Winnipeg, Canada

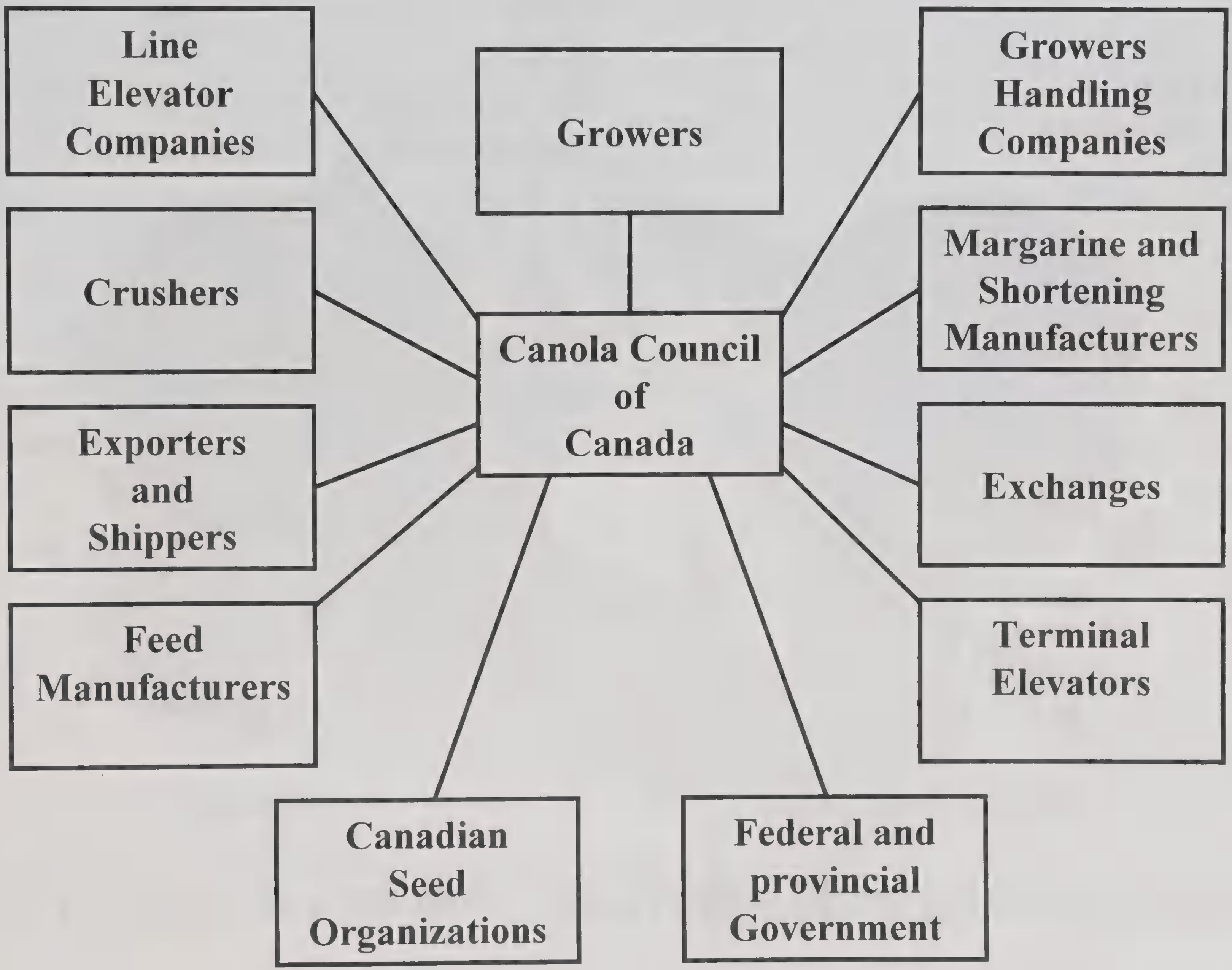
Key Points:

- **The primary markets for canola and canola products are Canada, the USA, China, Japan and Mexico - markets still considered essentially friendly to genetic engineering.**

- **Those markets are more concerned about GMO proteins than oils so the impact on canola has been less than on soy and corn as no segregating occurs once the product is commercialized.**

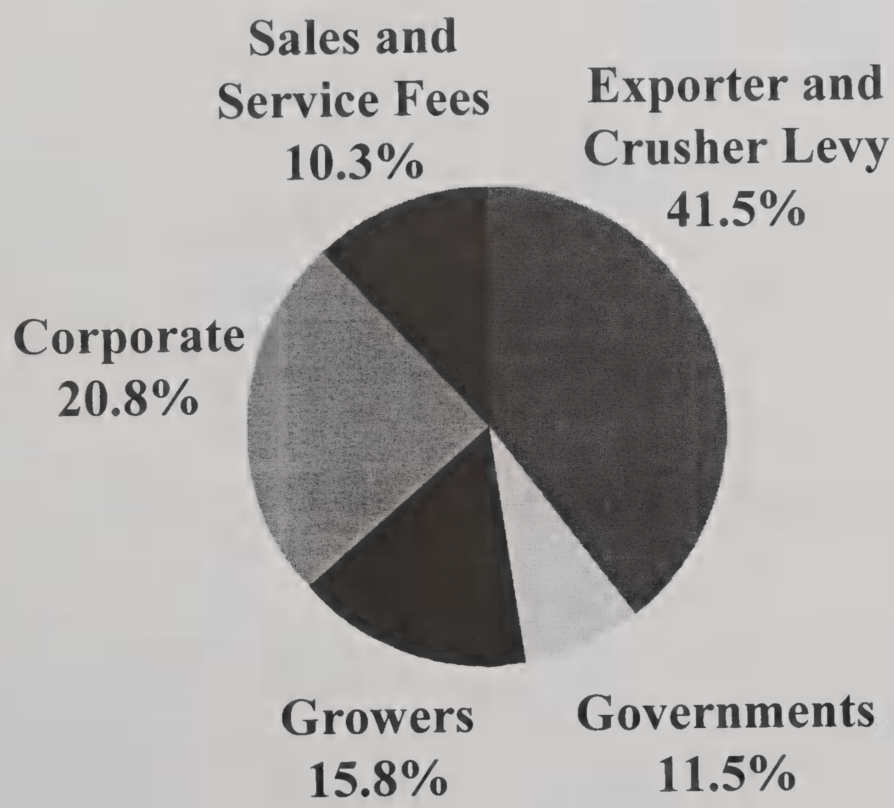
- **In terms of food safety, whether related to pesticides, E. Coli, or GMOs, consumers are moving away from a satisfaction with "no evidence of risk" to an apparent demand for "evidence of no risk".**

CANOLA COUNCIL OF CANADA

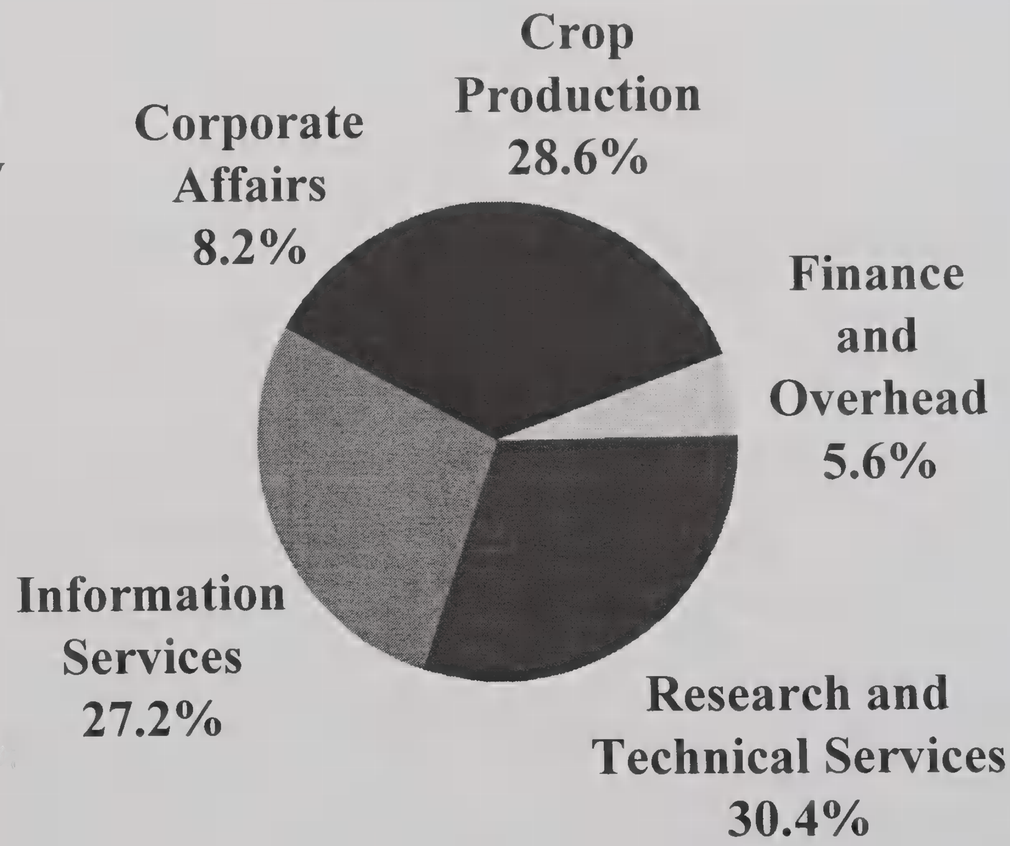


Canola Council of Canada - 2000 Operating Budget ($5,024,195)

Revenues

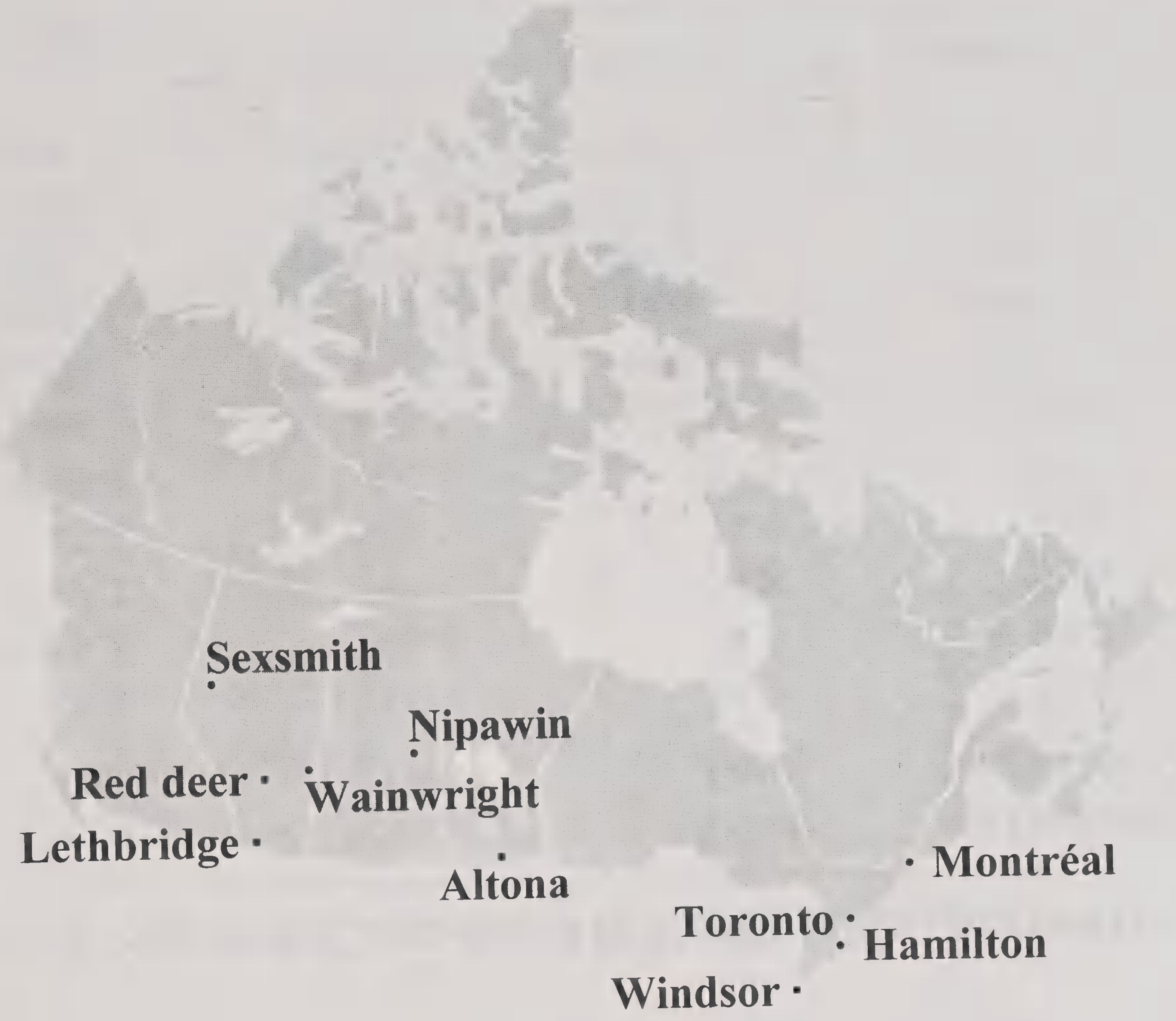


Expenditures



Canadian Crushing Plant locations

Canadian Refining Plant locations



Canola Share in Canadian Oil Production (percent)

	Margarine Oils	Shortening Oils	Salad Oils	All Deodorized Vegetable Oils
1976	32.8	16.2	50.2	31.0
1981	36.2	46.4	70.5	50.7
1986	38.2	49.4	79.8	58.3
1991	47.3	59.4	79.1	64.6
1996	48.8	64.3	87.5	75.4
1998	43.7	55.3	88.8	74.2

Japan: Domestic Production of Edible Oil (percent)

	Soyoil	Canola oil	Others
1976	45.3	26.4	28.3
1981	43.9	33.7	22.4
1986	42.4	36.6	21.0
1991	36.6	45.0	18.4
1996	37.3	44.5	13.4
1998	36.5	47.8	15.7

Canola Seed Demand & Supply

	1994/95	1995/96	1996/97	1997/98	1998/99	1999/00
Beginning stocks	330	589	1,030	563	363	633
Production	7,233	6,436	5,062	6,393	7,643	8,798
Imports	42	97	103	141	157	1227
TOTAL SUPPLY	7,605	7,122	6,196	7,096	8,164	9,553
Exports	3,912	2,804	2,519	2,964	3,900	3,892
Domestic crush	2,513	2,753	2,712	3,239	3,062	2,983
Seed, feed and waste	590	641	401	532	568	612
TOTAL DEMAND	7,015	6,198	5,632	6,735	7,530	7,487
ENDING STOCKS	589	990	563	363	633	2,066
STOCKS/USE	8.4%	16.9%	10.1%	5.4%	8.1%	27.5%

Canola Seed Exports (Thousand tonne)

	1995/96	1996/97	1997/98	1998/99	1999/00
Japan	1,678.5	1,734.4	1,829.4	1,814.5	1,800.9
United States	271.6	265.2	391.2	277.7	287.5
Europe	322.2	162.5	11.4	0.5	0.5
Mexico	530.8	355.8	593.0	529.2	570.1
China	--	--	110.1	1,269.2	1,211.1
Others	0.8	1.5	28.6	9.0	21.9
TOTAL	2,803.8	2,519.4	2,963.7	3,900.1	3,892.0

Canola Oil Exports (Thousand tonne)

	1994/95	1995/96	1996/97	1997/9	1998/99 e
United States	368.0	390.1	424.4	418.8	409.8
India	2.2	8.5	5.6	14.6	0.1
Hong Kong	1.1	35.4	85.7	156.1	94.6
China	28.1	36.9	42.5	71.2	121.6
Others	24.0	39.1	83.0	178.4	50.0
TOTAL	423.4	510.0	641.2	839.1	676.1

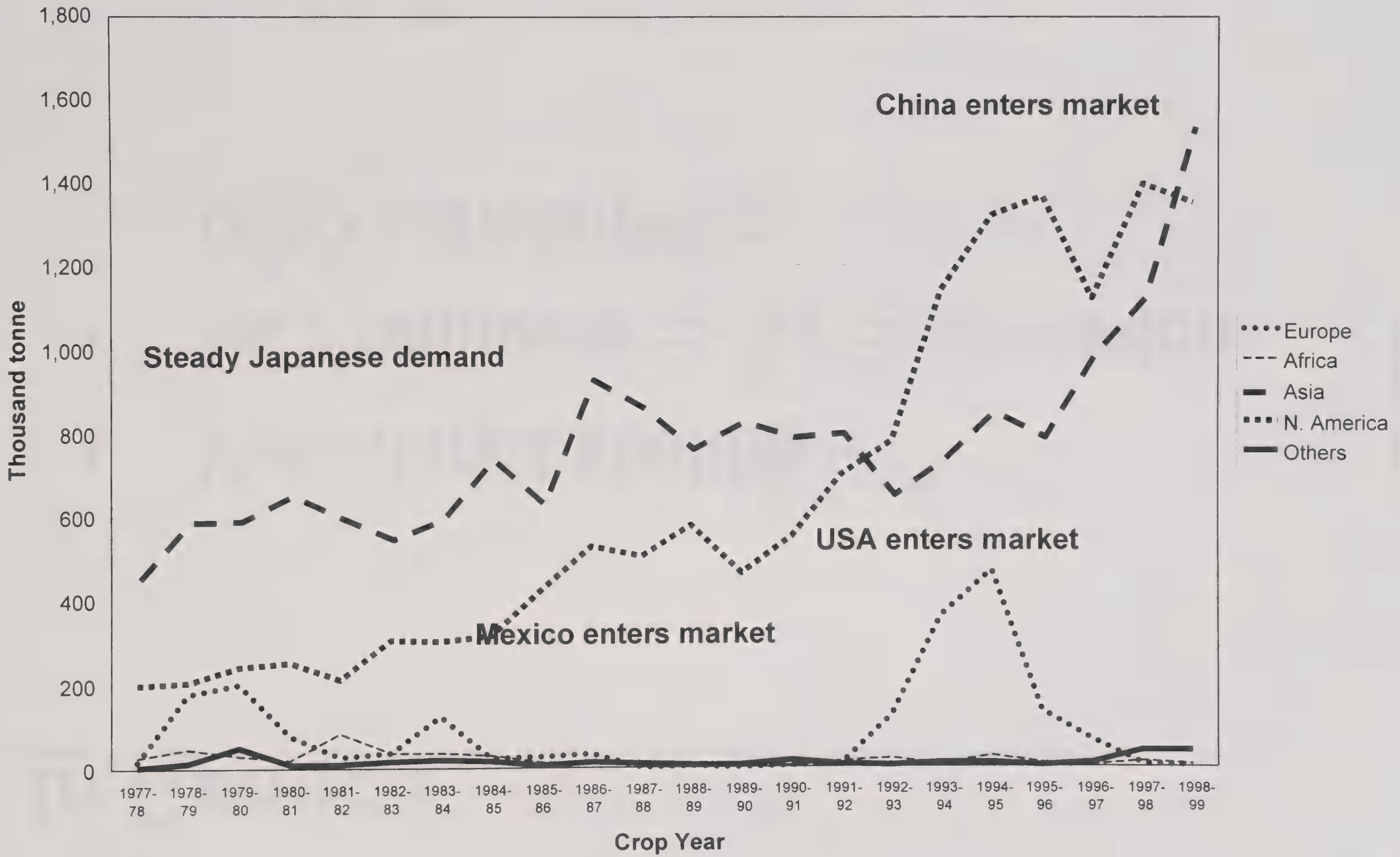
Canola Meal Exports & Total Utilization (Thousand tonne)

	1994/95	1995/96	1996/97	1997/98	1998/99
Europe	83.3	54.9	40.8	20.0	25.5
Japan	142.8	111.1	118.2	26.3	23.8
Indonesia	11.9	31.9	--	11.1	--
Taiwan	5.0	5.8	4.8	0.8	10.7
South Korea	55.4	71.6	34.9	49.8	56.2
United States	736.8	874.3	849.3	1,223.1	1,134.5
Others	32.3	54.6	36.9	88.0	8.1
Total Exports	1,067.5	1,204.2	1,084.9	1,419.1	1,258.8
Domestic Use	496.5	511.8	564.1	587.0	681.2
Total Utilization	1,564.0	1,716.0	1,649.0	2,004.0	1,940.0

Genetically Engineered Canola

- Round-Up Ready™
- Liberty Link™
- Invigor™ Hybrids
- Compas/Navigator™

Canola Demand in Oil Equivalents - By Continent



In Canada

- Voluntary Labeling
- No Premiums $\Rightarrow$ No Segregation
- GMO canola policy

In Japan

- **Labeling**
- **Illegal Imports**

UN Bio-safety Protocol

- LMO - living, for processing, inert
- Central Clearing House
- Liability & Responsibility

PARADIGM SHIFTS

- in variety development
- in pesticide development

PARADIGM SHIFTS

- Before = no evidence of risk
- Now = evidence of no risk

50th Oilseed Conference

Tools for Improving Profits

January 21–23, 2001 ✵ Hyatt Regency New Orleans

National Sunflower Association

Larry Kleingartner
National Sunflower Association
Bismark, ND

Key Points:

- **The US Sunflower industry is confident about the transition to NuSun.**
- **Major concern is competing for acres among a number of transgenic crops that present real production advantages to producers.**
- **Because of its excellent qualities, NuSun oil must generate more value than traditional sunflower for the producer, thereby keeping sunflower a viable cropping choice.**

National Sunflower Association

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50th Oilseed Conference: Tools for Improving Profits

Presentation by: Larry Kleingartner, National Sunflower Association (NSA)

Historically, the US sunflower industry has been dependent on exports of either sunflower seed or sunflower oil as its major market outlet. Up to 85 percent of the acreage was exported in one form or another. That heavy dependence became a real concern after the Uruguay Trade Round curtailed export subsidies. During the 90s many importing countries changed their purchasing from a central government buying agency to the private sector. Under the old system, a number of North African countries had a preference for either cotton or sunflower oil and would pay sizeable premiums to purchase these oils. With the private sector in control, price became the over riding purchase factor and the price premium, especially for sunflower oil, declined considerably.

Beginning in 1995, the sunflower industry looked for domestic opportunities. The best option appeared to be in the frying sector as a replacement for hydrogenated oils. This was elevated with the increasing concern about trans labeling issues. The concept of a mid-oleic (NuSun)* sunflower oil for this market segment was pursued with the NSA membership. The challenges of redirecting an entire industry are considerable. The major challenge is the production of mid-oleic hybrid seeds that farmers can depend upon to provide the same yield and oil content of their traditional hybrids. Another challenge is the segregation of NuSun seed in the market place. A final issue is consumer acceptance. Most of the challenges have been met. The hybrids are yielding well, inexpensive tests have been developed to determine NuSun in the market place, and consumers have found the oil to be functional in frying situations while providing an excellent taste profile.

Estimates of NuSun oil production are 120 million pounds in 1999-00, 340 million pounds in 2000-01 and we are anticipating a production level of about 500 to 600 million pounds in 2001-02. There are major efforts under way to convert the majority of the acreage to NuSun as soon as possible. The incentive for producers to change is high yielding hybrids and a price premium of about 10 to 13 percent over traditional sunflower.

However, the challenges are not over for the sunflower industry. The real challenge is yet ahead. That is to stabilize and grow the US acreage base. The world vegetable oil market is presently at a large surplus and protein dominates the value of the oilseed complex. That puts a high oil content seed like sunflower at a considerable

disadvantage to soybean in attracting acres. Plus, farmers in the Midwest are finding many agronomic advantages to growing Roundup Ready transgenic crops. Sunflower is a North American native species plant making it a poor choice as a Roundup Ready crop. There are regulatory concerns about cross-pollination to wild species. Weed control is a major limiting factor in growing sunflower, especially under reduced tillage scenarios.

To be successful in maintaining acres, NuSun oil must recapture that price premium that it had in past years. The market must 'price segregate' NuSun because of its functional properties as, not needing hydrogenation, its excellent taste profile, its low saturates and its non-transgenic status. Producers will need an incentive of about 20 percent over traditional sunflower prices to compete with soybeans and other transgenic crops for acres. The existing price incentive is already 10 percent or greater. At the same time, NuSun oil must remain competitive for major users. The original goal of the NuSun steering committee was that NuSun oil must to be priced as a commodity oil. That goal has not changed. However, that does not mean having to be a least cost oil. The sunflower industry is reasonably confident that the oil market will help make this transition thus providing the necessary incentives to the sunflower producer.

KEY POINTS

1. The US sunflower industry is confident about the transition to NuSun.
2. Major concern is competing for acres among a number of transgenic crops that present real production advantages to producers.
3. Because of its excellent qualities, NuSun oil must generate more value than traditional sunflower for the producer, thereby keeping sunflower a viable cropping choice.

* NuSun is defined as having the following fatty acid range:

Oleic – 55 to 70%
Linoleic – 20 to 35%
Saturates – under 10%

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The Cottonseed Oil Outlook

Lynn Jones
National Cottonseed Products Association
Memphis, TN

The Cottonseed Oil Outlook

Lynn Jones

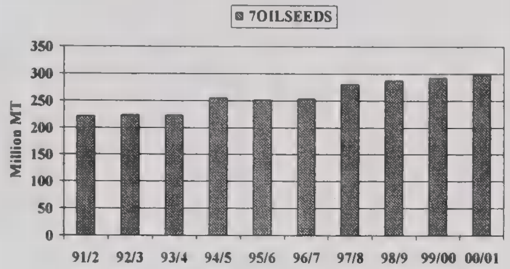
National Cottonseed Products Association

Memphis, TN

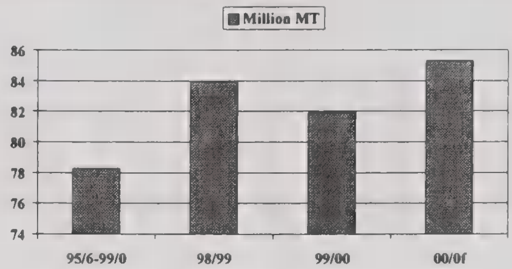
January 22, 2001

50th OILSEED CONFERENCE

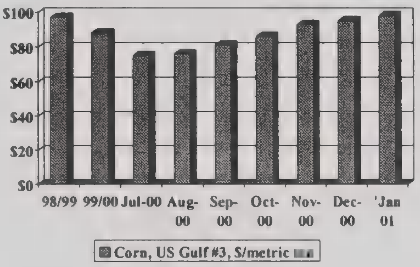
WORLD PRODUCTION OF 7 MAJOR OILSEEDS UP 34% IN 1990's (Oil World data)



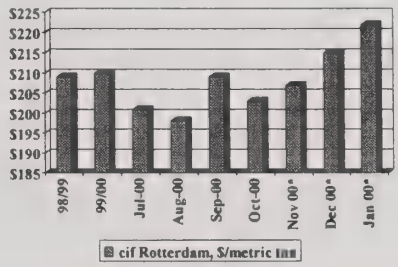
U.S. IS THE WORLDS LARGEST SINGLE OILSEED PRODUCER (Oil World 24Nov2000)



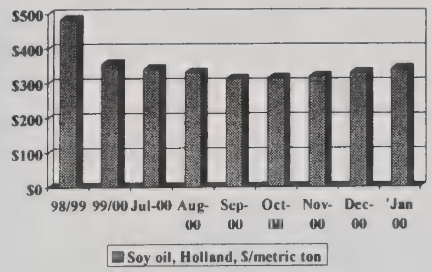
Corn prices have risen since mid-2000 (OW, 24 Nov 2000)



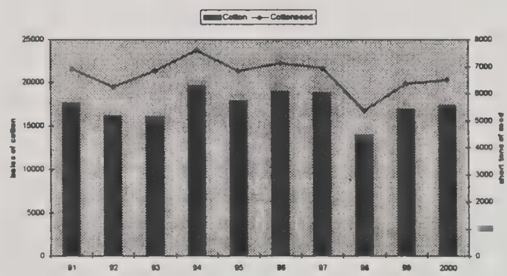
The price of soybeans has recently gone up. (OW 11/24/00)



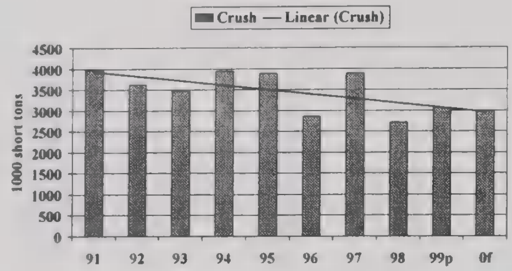
While soybean oil reflects flat oil prices (OW, 24 Nov 00)



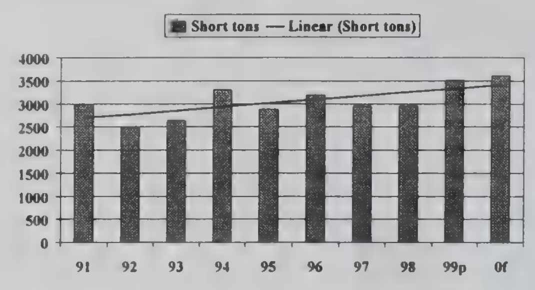
U.S. COTTON & COTTONSEED PRODUCTION (USDA)



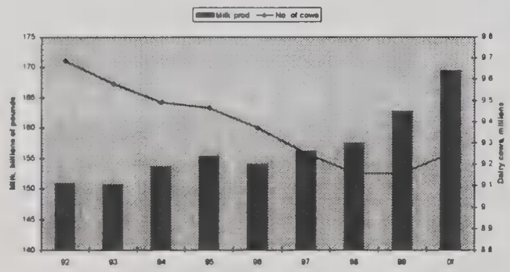
COTTONSEED CRUSHED (USDA)



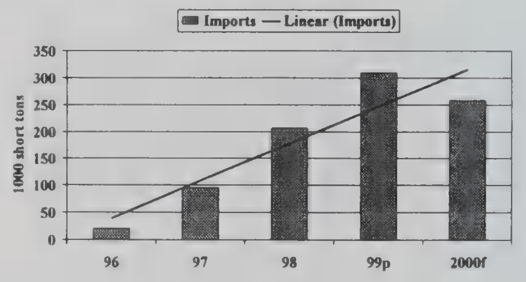
USDA's "*FEED, SEED & RESIDUAL*" REFLECTS COTTONSEED FED (USDA)



LOW FEED COSTS, HIGH MILK PRICE IN '98-9 ONLY PART OF CURRENT PICTURE (USDA)



IMPORT OF COTTONSEED IS A RECENT DEVELOPMENT



COTTONSEED OIL FITS

- Functionality - beta prime crystal structure= }shortenings, icings, plastic fat products
- Functionality - could avoid hydrogenation & labeling stigma in some uses
- Functionality - shelf life, taste, character, "brings out the taste"
- Availability - domestically produced with amount and price in predictable range

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MACT Regulation Update

Ron Moeller
Cargill
Minneapolis, MN

Material not available at press time.

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Future of Drought Resistant Oilseed (Soybean) Cultivars

Thomas E. Carter
USDA, North Carolina State University
Raleigh, NC

Material not available at press time.

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Extrusion-Expelling and Minimal Oil Refining for Processing Identity-Preserved Soybeans

Tong Wang* and Lawrence A. Johnson
Iowa State University
Ames, IA

Key Points:

- **Extrusion-Expelling is an alternative and unique soybean processing method**
- **Soybean oils and protein meals produced by extrusion-expelling have different quantities than those produced from solvent extraction**
- **Soybean oil obtained from this processing could be minimally refined to produce specialty products for the niche market**

Extrusion-Expelling and Minimal Oil Refining for Processing Identity-Preserved Soybeans

Tong Wang and Lawrence A. Johnson
Iowa State University

50th Oilseed Conference, January 21-23, 2001

Extrusion-Expelling (E-E), developed in the 1960s to enable farmers to cook their soybeans for use as livestock feed, has been increasingly used for value-added soybean processing. In E-E processing, dry extrusion is used as a heat pretreatment and oil is removed by an expeller press. It is an unique process in which less capital investment, less energy, and small seed tonnage are required compared to conventional solvent extraction (SE). In addition, the process is mechanical and there is no environmental consequence because flammable solvent is not used. E-E is particularly suitable for small quantities of various types of soybeans, such as non-GMO, organic or natural, genetically enhanced, and other identity-preserved seeds. To identify more value-added opportunities for E-E oil and meal, we compared the quality of oils and meals produced from E-E and SE, evaluated the effect of E-E processing condition on oil and meal quality, and developed refining method with which the E-E oils could be naturally and minimally refined.

Comparison of Qualities of Oils and Meals Obtained by E-E and SE

A survey we conducted with 13 E-E mills and 9 SE plants showed that soybean oils and meals produced by E-E differed from those produced by SE, as summarized in Tables 1 and 2.

Table 1. Comparison of Extruded-Expelled (E-E) and Solvent Extracted (SE) Oil

	E-E		SE	
PV, meq/kg	1.73	a*	0.95	b
FFA, %	0.21	b	0.32	a
Phosphorus, ppm	79	b	267	a
AOM stability, hr	24.3	b	40.8	a
Tocopherols, ppm	1257	b	1370	a
Color, Red	10.2	a	11.1	a

* Values in the same row with different letters are significantly different at 5%.

PV of E-E oil was significantly higher than that of the SE oil, which we attribute to poor oil storage conditions and longer storage time at E-E mills and/or the high temperature used in E-E process. FFA content of E-E oil was significantly lower than that of SE oil, which may be due to the rapid denaturation of the catabolic enzymes during extrusion.

Phospholipid (PL) content of the oil after natural settling was much lower in E-E oil than in SE oil. PLs in E-E oil are more hydratable which we attribute to the rapid heat denaturation of the phospholipases. Total tocopherol content of the E-E oil was slightly, but significantly lower than that of the SE oil. Oxidative stability, as measured by AOM (Active Oxygen Method), of the E-E oil was significantly lower than that of the SE oil, probably due to higher PV and lower phosphorus and tocopherol contents of E-E oil. Colors of the two oils were not different.

Table 2. Comparison of Extruded-Expelled (E-E) and Solvent Extracted (SE) Meal

	E-E		SE	
Urease, ΔpH	0.07	a*	0.04	a
KOH solubility, %	88.1	a	89.1	a
PDI, %	18.1	b	44.6	a
Oil, %[a]	7.2	a	1.2	b
Protein, %[a]	42.5	b	48.8	a
Fiber, %[a]	5.4	a	3.7	b
Rumen bypass, %	37.6	a	36	a
Trypsin Inhibitor, TIU/g	12254	a	5275	b

* Values in the same row with different letters are significantly different at 5%.
[a] Percentages are based on 12% moisture content.

The degree of protein denaturation of soybean meal is measured by protein solubility under alkaline (KOH) conditions, urease activity, and protein dispersibility index (PDI). KOH solubilities of E-E and SE meals were not significantly different, neither were urease activities. PDI values of E-E meals were much lower than those of the SE meals, indicating higher degrees of protein denaturation. The ruman bypass values were similar for E-E and SE meals. Residual oil, protein, and fiber contents were different in the two meals due to no-dehulling and high residual oil of E-E meal. Trypsin inhibitor was reduced to a much lower level in SE meal than in E-E meal.

Effect of E-E processing on Oil and Meal Quality

Various processing conditions were used to produce E-E protein meals with different degree of heat denaturation and residual oil content, and the effect of heat on oil quality was evaluated (Figs 1, 2, and 3). As indicated in the figures, the more the oil was removed, the more severe the protein was heat denatured, the more free fatty acid and phospholipid the extracted oil contained, and the darker the oil became. Oil oxidation was not affected by processing conditions as much as the other parameters.

Fig1. Relationship of Protein Denaturation and Residual Oil Content of E-E Meal

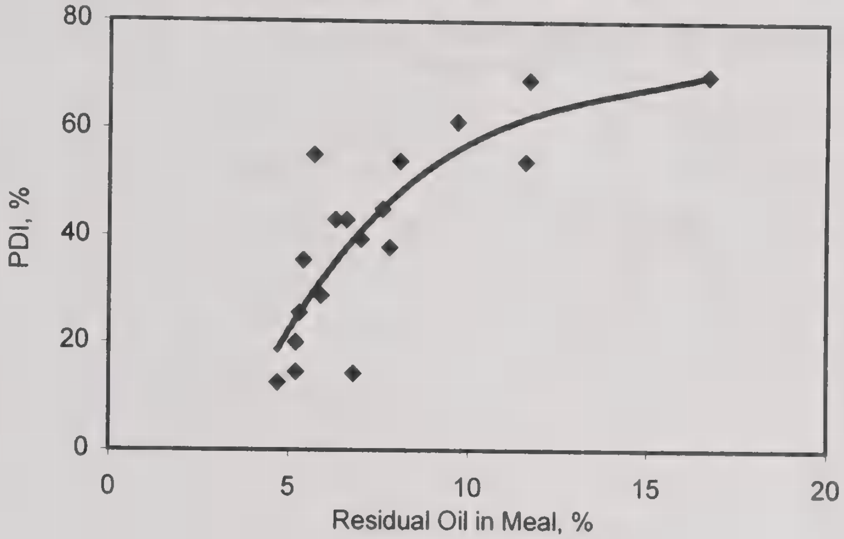
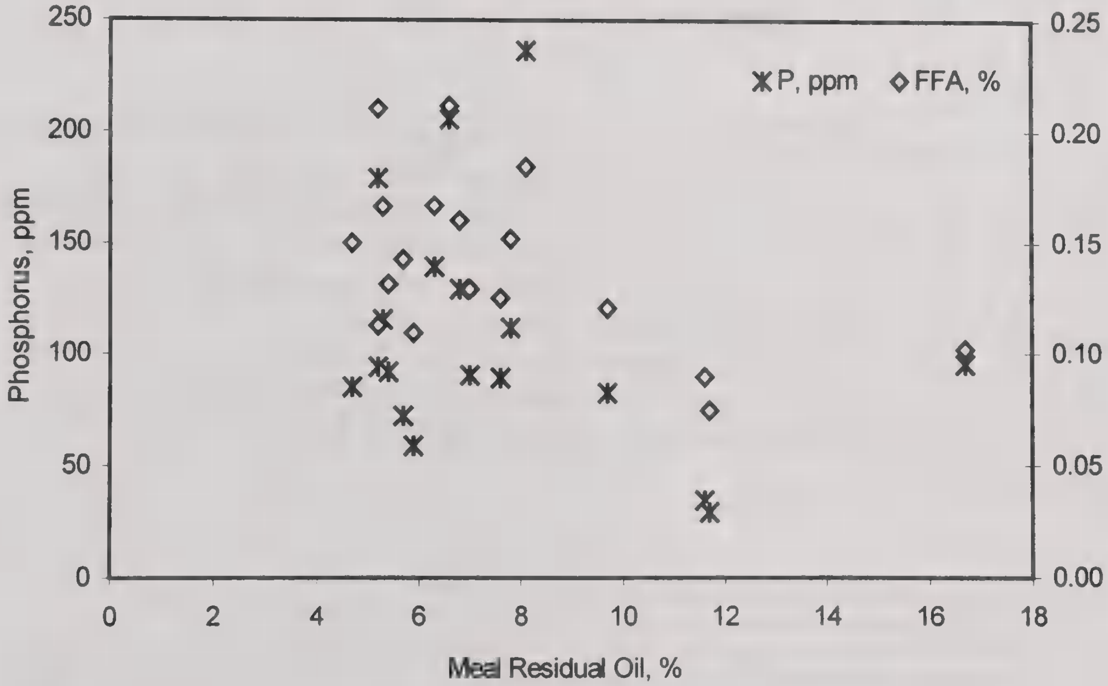


Fig 2. Phospholipid and Free Fatty Acid Contents as Affected by Meal Residual Oil Content

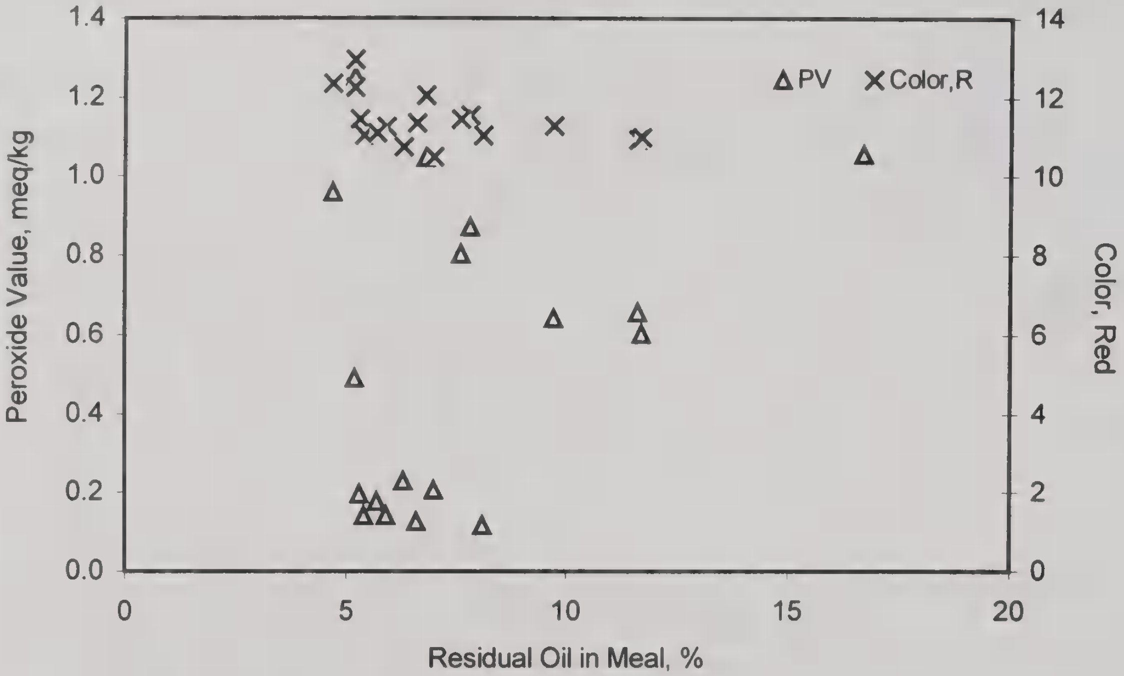


Minimal Refining of E-E Oils with Modified Fatty Acid Compositions

Simple, low-capital-investment oil refining techniques, which may also meet the needs of natural or organic food industries, were explored to process E-E soybean oils with various fatty acid compositions (Table 3 shows the fatty acid compositions of the oils tested). Most settled E-E oils contained low level of phospholipids (< 400 ppm phosphorus) and were easily water degummed to low phosphorous levels (< 55 ppm).

Free fatty acid was reduced to less than 0.04% by an adsorption treatment with 3% Magnesol®.

Fig 3. Oil Oxidation and Free Fatty Acid Content as Affected By Meal Residual Oil Content



Magnesol reduced residual phosphorus contents to negligible levels. This material also adsorbed primary and secondary oil oxidation products. Silica was used to further reduce the levels of oxidized lipids. As shown in Fig 4, the adsorption refining procedure was much milder than conventional refining, as indicated by little formation of primary and secondary lipid oxidation products and less reduction in tocopherols. More unsaturated oils oxidized more easily during refining than did the other types of oils. High-oleic soybean oil had excellent oxidative stability and better flavor characteristics after refining compared with other oils. This research showed that E-E oils could be refined by this minimal processing method with less damage to the oils compared with conventional method.

Table 3. Fatty Acid Compositions of Modified Soybean Oils

	16:0	18:0	18:1	18:2	18:3
CS	10.8	4.9	25.2	51.6	7.5
LOX	10.2	4.6	33.1	45.4	6.7
HO	6.7	3.8	79.2	7.1	3.1
LS	4.6	3.8	22.4	62.0	7.1
LLL	10.7	4.6	25.0	56.6	3.1

Abbreviations: CS, commmercial soybean oil; LOX, lipoxygenase-free soybean oil; HO, high oleic acid soybean oil; LS, low saturated fatty acid soybean oil; and LLL, low linolenic acid soybean oil.

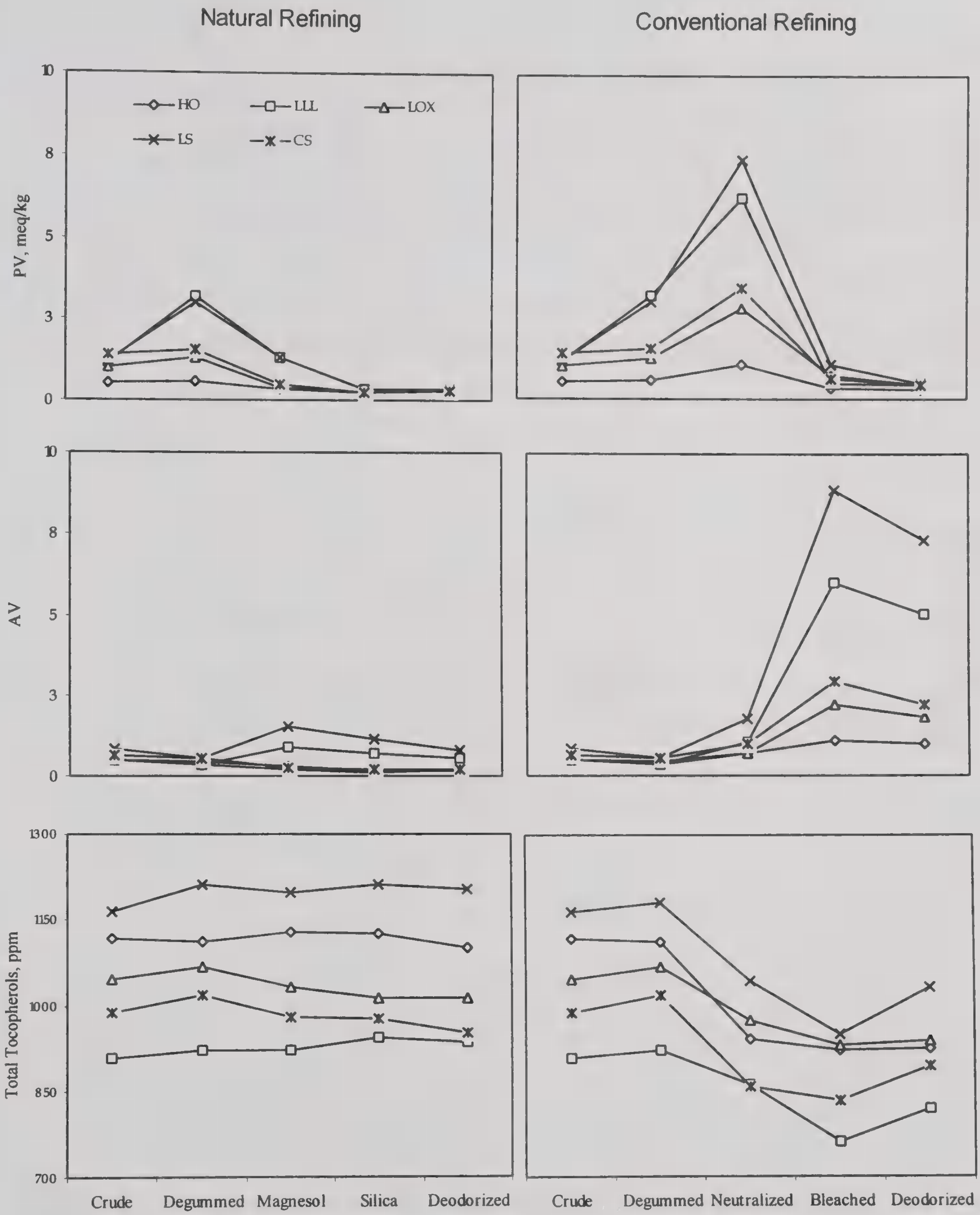


Fig 4. Refining characteristics of E-E oils with modified fatty acid compositions
HO - High oleic acid; LLL - Low linolenic acid; LOX - Lipoxygenase-free; LS - Low saturated acids; CS - Commercial Soy

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Three Main Points

1. Extrusion-Expelling is an alternative and unique soybean processing method;

2. Soybean oils and protein meals produced by extrusion-expelling have different quantities than those produced from solvent extraction;

3. Soybean oil obtained from this processing could be minimally refined to produce specialty products for the niche market.

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Soapstock Utilization: A Process for the Production of Biodiesel Fuel

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Soapstock Utilization: A Process for the Production of Biodiesel Fuel

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New Orleans, LA

Abstract

'Biodiesel' is the term given to simple alkyl esters of fatty acids intended for use in compression ignition (diesel) engines in place of petroleum-derived fuel. Compared with the latter, it offers several attractive features, among them pollution reduction and a measure of national energy independence. Highly refined vegetable oils were the original feedstock for biodiesel production. However, their high costs can render the resulting product so expensive that it cannot compete economically with petroleum-derived fuel. This has stimulated investigation of the use of alternate, cheaper, feedstocks for biodiesel production. We have developed a method for the production of fatty acid methyl esters from vegetable oils soapstock, an inexpensive coproduct of vegetable oil refining, and report here the results of engine emissions testing of this fuel in a production model heavy-duty diesel engine (11.1 liter displacement). Compared with petroleum diesel fuel, emissions of total hydrocarbons, particulates, and carbon monoxide were reduced 55%, 53% and 48%, respectively, during operation on neat soapstock biodiesel. Nitrogen oxides increased 9%, and carbon dioxide was elevated 3.7%. During operation on a 20 vol % blend of soapstock biodiesel in petroleum diesel, reductions of 27.7%, 19.7%, and 2.4%, respectively, in total hydrocarbons, particulate matter, and carbon monoxide were noted. Nitrogen oxide and carbon dioxide emissions were increased by 1.3% and 1.2%.

INTRODUCTION

Biodiesel is the name given to the monoalkyl esters of animal- or vegetable-derived long chain fatty acids when intended for use as replacements for petroleum-derived fuel in diesel engines. Extensive research conducted worldwide,[1-4] especially in the past two decades, has resulted in: a)

the development and optimization of methods for the synthesis of biodiesel from a variety of lipids, b) characterization of the performance and exhaust emissions of engines fueled with biodiesel,[5] and c) investigation of the toxicity of these emissions. These studies have established the suitability of biodiesel from a performance standpoint, and its attractiveness in terms of enhanced fuel lubricity, reduced emission of regulated air pollutants, and the reduced toxicity of those pollutants that are emitted.

Biodiesel reduces emissions of carbon monoxide (CO), hydrocarbons (HC), mutagens, air toxics and particulate matter (PM) relative to petroleum diesel, but can slightly increase levels of oxides of nitrogen (NO and NO_2, collectively termed NO_x).[5] Since emissions depend on fuel chemical composition, they are feedstock-dependent. Determination of the emissions profile is a key step in assessing the acceptability of a biodiesel fuel produced from a new feedstock.

A major barrier to the use of biodiesel has been its cost. When made from refined oils, approximately 70% of the product cost can be due to the expense of the feedstock. This fact has triggered interest in the use of lower value lipids in biodiesel production.

Soapstock (SS) is a relatively inexpensive byproduct of edible oil refining, and is rich in the fatty acids that are the precursors of biodiesel. We have previously developed and optimized a method for the synthesis of fatty acid methyl esters (FAME) from soybean soapstock.[6] We describe here the emission properties of the resulting fuel in a representative diesel engine operated according to Federally-specified test conditions.

MATERIALS AND METHODS

Chemicals. Soybean SS was a gift from Cargill Inc., Gainesville, GA. Biodiesel produced from refined soy oil (Soygold) was the product of Ag Environmental Products L.L.C. (Lenexa,

KS). Reference (certification grade) petroleum diesel fuel was obtained from Phillips Petroleum (Borger, TX). Other materials were obtained from standard chemical supply houses.

Synthesis of Fatty Acid Methyl Esters. Esterification of the fatty acids in SS was achieved by first conducting an alkaline hydrolysis of all fatty acid ester bonds, followed by lyophilization to remove water, and sulfuric acid-catalyzed methyl esterification of the resulting free fatty acids.[6] This protocol yielded about 2.5 L of methyl ester per 5 kg of dried saponified SS. Through successive reactions, 25 liters of fatty acid methyl ester was accumulated. High performance liquid chromatography[7] (HPLC) and gas chromatography[8] (GC) were employed in this laboratory to measure the degree of esterification and to identify and quantitate the fatty acid methyl esters in the product. Additional compositional and analytical testing was conducted by Williams Laboratory Services (Kansas City, KS).

Emissions Measurement. Engine emissions were measured according to U.S. Federal specifications for heavy-duty engine emissions certification testing[9] as described previously.[10] The test engine was a 1991 calibration, 11.1 liter displacement, six cylinder Detroit Diesel heavy duty engine, nominally rated at 345 bhp (257 kW) at 1800 rpm.

RESULTS AND DISCUSSION

Production of Biodiesel from Soapstock. The method employed here for FAME synthesis from soapstock involved (*i*) hydrolysis of all fatty acyl ester bonds by saponification, (*ii*) removal of water, and (*iii*) acid-catalyzed esterification of the resulting free fatty acids. The process was simple and efficient, involved readily available and inexpensive reagents, operated at ambient pressure and relatively low temperatures, and was not difficult to conduct.

The saponification treatment caused essentially complete hydrolysis of all acylester bonds in

SS, as assessed by HPLC (minimum detection limits: glycerides: <0.04%, phosphoglycerides and lysophosphoglycerides: <0.06%). The esterification reaction was also highly efficient, leaving less than 0.5% of the input free fatty acids unesterified. The recovery of crude FAME averaged 96% of theoretical maximum.

Characterization of Soapstock-Derived Biodiesel. The fatty acid composition of the FAME product was essentially identical to that reported for soybean SS,[11] and similar to that of biodiesel produced from refined soy oil.[12] Reflecting differences in the fatty acid contents of the raw materials from which they were made, soapstock methyl ester contained about 50% more palmitic acid than did soy oil-based biodiesel, and a correspondingly lower amount of oleic acid.

The other predominant fatty acids in these preparations (oleic, linoleic, linolenic) were present at comparable levels in FAMEs produced from soy oil and soapstock.

Compositional and physical properties of the SS-derived FAME were compared with the Provisional Standard for biodiesel published by the American Society for Testing and Materials (ASTM). The material was within specifications for all assayed variables. Among these were ester content (99.2%, vs. a specified minimum of 97.0%), acid no. (0.05 mg/gm, vs. 0.80 mg/gm maximum), free glycerin (0.00%, vs. 0.02 % max.), total glycerin (0.12%, vs. 0.24% max.), and water (<0.01%, vs. 0.05% max.).

Engine Emissions. Emissions data were expressed relative to comparable data obtained during engine operation on emissions-certification grade petroleum-derived diesel fuel (Table 1).

Relative to certification diesel, neat soapstock biodiesel increased NO_x emissions by 9% and decreased PM by slightly more than 50%. This NO_x emission level was not statistically different from that obtained with fuel produced from refined soy oil. The PM emissions for neat

SS biodiesel were significantly higher than for fuel made from refined soy oil, but were well within the range reported for soy-based biodiesels in similar engines.[5] Emissions of CO and hydrocarbons with SS biodiesel were generally comparable to those with fuel produced from refined soy oil, though the reduction in CO was not as great at the B20 level for SS biodiesel as for soy biodiesel.

Because biodiesel is frequently used as a 20 volume percent blend with conventional diesel (so called B-20), the SS methyl ester fuel was also tested as a B-20 blend. This blend produced a small, though statistically significant, 1.5% NO_x increase, and a 20% PM reduction, relative to certification diesel. These emissions levels are comparable to emissions of soybean oil methyl ester B-20.[5]

Taken together, these data indicate that from the standpoint of engine emissions an acceptable diesel fuel can be synthesized from vegetable oil soapstock.

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TABLE 1: Emissions of Federally Regulated Pollutants by a Heavy Duty Diesel Engine Operating on Neat (B100) and 20% Blends (B20) of Soy Oil- Derived and Soapstock-Derived Biodiesel[a]

Property	Fuel			
	Soy Oil Biodiesel		Soapstock Biodiesel	
	B100	B20	B100	B20
Total Unburned Hydrocarbons	- 93%	- 30%	- 55%	- 28%
Carbon Monoxide	- 50%	- 20%	- 48%	- 2.3%
Particulate Matter	- 30%	- 22%	- 53%	- 20%
Nitrogen Oxides	+ 13%	+ 2%	+ 9%	+ 1.3%

[a]Data are expressed relative to the emissions obtained with Certification Grade petroleum diesel fuel.

50th Oilseed Conference

Tools for Improving Profits

January 21–23, 2001 * Hyatt Regency New Orleans

Value-Added Adsorbents from Oilseed Hulls

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New Orleans, LA

Key Points:

- **A process was developed to enhance metal ion adsorption in soybean hulls**
- **Modified soy hulls adsorb metal ions as well as commercial resins**
- **Modified soy hulls can be produced at a relatively low cost compared with commercial resins**

VALUE-ADDED ADSORBENTS FROM OILSEED HULLS

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Oilseed hulls, in particular soybean hulls, have inherent adsorbent properties, especially toward metal ions. Our laboratory has developed a method to enhance metal ion adsorption by soybean hulls. Hulls were modified in the presence of citric acid to produce adsorbents with a high adsorption capacity for several metal ions, including cadmium, copper, lead, nickel and zinc.

Select properties of the modified soybean hulls were compared to similar properties of commercial chelating or cation exchange resins. The physical property of particle integrity (attrition) was evaluated. Modified hulls were found to have similar attrition as commercial resins except at temperatures greater than 65°C, where attrition was higher in the modified hulls. The property of metal ion adsorption was also investigated. Our comparative results with commercial resins demonstrate that modified soybean hulls have similar metal ion adsorption characteristics and can be considered a product with potential commercial application for metal ion remediation.

Our laboratory has also optimized the citric acid modification process and a description of the process, along with capital, manufacturing and product costs are presented.

Introduction

Oilseed hulls, including soybean hulls, constitute a low value product sold to animal feed formulators. Because of their great abundance (about 10 billion lbs produced in the United States each year) and low value, additional, higher value product outlets need to be identified. In recent years, attention has focused on the utilization of soybean hulls as an adsorbent, particularly for metal ions (Laszlo, 1987; Marshall and Champagne, 1995; Marshall and Johns, 1996). Although hulls have been shown to bind various metal ions, their metal ion adsorption capacity was inferior to commercial resins.

In order to improve metal ion adsorption in soybean hulls, Marshall et al. (1999) washed hulls with sodium hydroxide, then modified them with a food grade acid, citric acid (CA). Adsorption of copper ion (Cu^{2+}) was greatly improved over unmodified hulls. Moreover, when the use of CA-modified hulls was extended to include other metal ions considered environmental pollutants, namely, cadmium, lead, nickel and zinc, the modified hulls readily adsorbed these metal ions (Marshall et al., 2000). A comparison of commercial resins and modified hulls resulted in little difference in metal ion uptake.

Although CA-modified hulls have been shown to adsorb metal ions, they should have the requisite physical properties to be able to withstand use in both batch and

column applications. The physical property most readily visible during use of an adsorbent is attrition. Attrition is defined as 'the unwanted breakdown of a particle within a process' (Bemrose and Bridgewater, 1987). The physical integrity of the modified hulls was compared the physical integrity of two commercial resins (Marshall et al., 2000). Marshall et al. (2000) demonstrated that in terms of product integrity, CA-modified soybean hulls could be useful in several batch and column applications requiring a metal ion adsorbent, but not necessarily at temperatures above 65°C.

The soybean hull modification process developed by Marshall et al. (1999) was optimized in preparation for possible production of the modified product (Marshall et al., submitted). This study defined the optimized process by means of a flow chart, determined capital, equipment and operating costs and gave an estimate of the production cost.

This report documents research conducted in our laboratory at the Southern Regional Research Center on technology to create value-added adsorbents from agricultural by-products. The remainder of this report will describe in further detail and discuss the results of studies from our laboratory to achieve that goal.

Results and Discussion

Improvement of metal ion adsorption by CA modification

In order to demonstrate that CA modification of soybean hulls improved their ability to adsorb metal ions (copper ion), unwashed hulls were used as the control and compared to base (sodium hydroxide)-washed hulls and base-washed hulls modified with CA after exposure to different concentration of CA. Adsorption capacities, which determine the maximum amounts of metal ion capable of being adsorbed by the hulls, were used for comparison purposes. Additionally, total negative charge for the hulls was also determined as a measure of the extent of modification. CA modification should add negative charge to the hulls, thus increasing the amount of metal ion bound. The results are given in Table 1.

Base-washed (BW) hulls and BW hulls modified after mixing with the lowest CA concentration (0.1 M) had higher adsorption capacities than unwashed (UW) hulls. However, UW hulls had a greater total negative charge. Therefore, BW samples had a total negative charge that was more efficient in binding copper ion with respect to copper ion uptake. Modification of hulls at progressively higher CA concentrations resulted in progressively higher adsorption capacities and total negative charge. Over the CA concentration range used, no maximum adsorption capacity or maximum total negative charge was observed. The adsorption capacity for the sample reacted with 1.2 M CA and subsequently modified was greater than that obtained using purified starch and starch components as a substrate for CA derivatization (Wing, 1996a) and also greater than that observed during derivatization of corn by-products with CA (Wing, 1996b; Wing, 1997). Therefore, soybean hulls appear to be a good feedstock for this type of modification and yield excellent metal ion adsorbents.

Comparison of metal ion binding with CA-modified hulls and commercial resins

On the basis these high adsorption capacities (Table 1), especially with exposure of the hulls to CA concentrations of 0.6 M to 1.2 M, we wanted to compare adsorption

values with commercial resins and additional metal ions. For this series of experiments, we chose BW hulls exposed to 0.6 M CA. Although Table 1 shows higher adsorption capacities at CA concentrations above 0.6 M, the cost of CA at these higher solution concentrations could preclude production of a competitive product for the commercial market.

BW, CA-modified soybean hulls were compared to commercial chelating and cation exchange resins for their ability to sequester five different metal ions (cadmium, copper, lead, nickel, zinc), either individually or as a group, from laboratory prepared solutions. The four commercial resins were chosen to represent both chelating (Amberlite IRC-718, Duolite GT-73) and strong cation exchange (Amberlite IR-122, Amberlite 200) resins. They were also chosen because of their affinity for the metal ions used in this study (Supelco, 1998).

Modified hulls and commercial resins were exposed to solutions containing a 7 mM concentration of metal ions. At this high concentration of metal ions, large differences in total amounts adsorbed among hulls and resins appeared and also many of the adsorbents clearly showed a preference for specific metals (Table 2). At total metal ion concentrations (35 mM) that saturate modified hull adsorption sites, modified soybean hulls adsorbed more metal ions than any of the commercial resins. The modified hulls exhibited a preference for Cu^{2+} and Pb^{2+}, but the other metal ions were removed at levels above 0.100 mmoles g^{-1}. In this situation, where all metal ions are competing with each other for limited adsorption sites, the order of preference was $Cu^{2+} > Pb^{2+} > Cd^{2+} > Zn^{2+} = Ni^{2+}$.

High adsorption capacity, specificity towards copper and lead ions, but good adsorption of cadmium, nickel and zinc ions, point to a modified hull preparation with promising commercial potential as a metal ion remediation product.

Attrition in CA-modified hulls and commercial cation exchange resins

We determined the attrition of soybean hulls that were base washed and subsequently modified with 0.6 M CA. Attrition values were determined under different experimental conditions of temperature and pH using a batch method and under continuous flow conditions in a column application.

Modified hull attrition was also compared to attrition in commercial cation exchange resins and the data presented in Table 3a. After a 24 hr incubation, modified hulls had the lowest percent attrition compared to both commercial products.

When the modified hulls and commercial resins were exposed to different temperatures, the modified hulls appeared to be most affected (Table 3b). At incubation temperatures of 25° and 45°C, modified soybean hulls showed significantly less attrition than IRC-718, and the same degree of attrition as IR-122. At incubation temperatures of 65 and 85°C, the modified hulls showed significantly higher attrition than both resins. Resin attrition was lower in Table 3b than in Table 3a. The difference is likely due to stirring with a stir bar (Table 3a) rather than shaking in a rotary shaker (Table 3b). This points out that different attrition methods can yield different results. The mechanical action of the stir bar appears more severe than the mechanical action of the shaker. However, comparisons within a specific set of test conditions should be valid.

The high attrition observed in modified hulls at 65°C and 85°C for 24 hr of stirring also lead to a diminution of copper ion (Cu^{2+}) adsorption by the hulls. Copper ion uptake was 1.54, 1.52, 1.20 and 0.91 mmoles Cu^{2+}/g hulls at incubation temperatures of 25°C, 45°C, 65°C and 85°C, respectively. No difference in copper ion uptake was observed between the samples incubated at 25°C and 45°C. The hulls exposed to temperatures of 65°C and 85°C bound 22% and 41% less copper ion, respectively, than the sample incubated at 25°C. Exposure of the modified hulls to high temperatures may hydrolyze some of the citric acid groups from the hull surface since a concomitant reduction in negative surface charge was also observed.

Attrition in modified hulls increased with increased pH as shown in Table 3c. Thus, the modified hulls appeared more stable under acidic conditions. Commercial cation exchange or chelating resins normally operate at acidic pH of 6 or less because many metal ions precipitate as hydroxides and are not removed by the resin at higher pH values (Lankford, 1990).

Incubation of modified hulls at different pH values had little effect on their subsequent ability to adsorb copper ion. Modified hulls that had been exposed to pH values of 3, 7 and 11 for the attrition studies, adsorbed 1.64, 1.62 and 1.53 mmoles Cu^{2+}/g hulls, respectively. Thus, modified hulls retain their negative surface charge after prolonged (24 hr) exposure to both acidic and alkaline pH. These results also suggest that little if any hydrolysis of citric acid groups occurred on the hull surface over the broad pH range of 3-11.

Modified soybean hulls and commercial cation exchange resins were subjected to a continuous flow of pH 4.8 buffer in a packed column over 72 hr period at a flow rate of approximately 10 ml/min. The mass of modified hulls removed from the column was 6.7%, which was lower than the 8.8% attrition obtained for IRC-718 but greater than the 1.9% attrition for the IR-122 resin. Since in many applications resins can remain in a column or be in contact with effluent flow for far longer than 72 hr, our column results show the potential value of the modified hulls and their actual value awaits further testing.

Optimization and estimated cost of production for CA-modified hulls

Based on the excellent results obtained for metal ion adsorption by modified hulls, we conducted studies to optimize this process. Once the process was optimized to our satisfaction, we then endeavored to define the process by means of a flow chart, determine capital, equipment and operating costs, and finally give an estimate of product cost.

Since we used BW hulls in earlier experiments, a comparison of adsorption capacities was used to determine the need for a wash step, be it base or water, before modification with CA. Adsorption capacity was used as a measure of the effectiveness of the presence or absence of a washing step in adsorbing copper ions. The adsorption isotherms for BW, WW, and UW, CA-modified soybean hulls were generated and adsorption capacities calculated from the isotherms are given in Table 4.

Analysis of the data indicate that there is no significant difference in adsorption capacity for the three different samples. The lack of a difference in copper ion adsorption may be accounted for by the similar total negative charge values among the

samples as depicted in Table 4. Therefore, a wash step was not considered essential and we recommend that hulls be used for CA-modification without prior washing.

In our original process, the hulls were dried at 50°C for at least 2 hr prior to modification at 120°C for 90 min in order to allow the reactive species, citric anhydride, to form from citric acid on the hulls. To eliminate a drying step and possibly increase product throughput, we compared a series of different modification conditions without the prior drying step to a control sample prepared by our original process. Copper ion adsorption efficiency was used as the indicator to evaluate the different drying conditions. The results are presented in Table 5.

The sample designated UW-A was subjected to initial drying prior to modification (post-CA soaking), but the series of samples designated UW-B through UW-D were not. These samples differed only by the length of exposure to the modification temperature of 120°C. Analysis of the data indicate that there were no differences between any of the treatments. Since the treatments were not significantly different, an initial drying step at 50°C is not needed and the modification time can be kept at 90 min.

Reaction efficiencies were also estimated for the samples in order to determine the amount of CA that could be recycled during washing of the CA-modified hulls (Table 5). A reaction efficiency of (27%) was determined for UW-B, which are the reaction conditions suggested for use. Therefore, theoretically, we should be able to recover (72%) of the unreacted citric acid for reuse.

Based on our original method and the new options derived from the data in Tables 4 and 5, the optimized citric acid modification process was described in a flow diagram given in Figure 1. Ten thousand kg/day of soybean hulls are fed to a milling unit, which consists of a grinder and a series of sieves. We estimate that an 80% yield of the 10 x 20 mesh fraction of soy hulls can be obtained by adjustment of screen size and grinder speed in the milling unit. The smaller particles (<20 mesh) are sent to a separate storage facility for further processing (e.g. as animal feed). The larger particles (>10 mesh) are recycled to the original storage facility and are eventually passed through the milling unit again. From the milling unit, the 10 x 20 mesh fraction of soybean hulls are conveyed directly to the CA soak tank. The soybean hulls are soaked in 0.6 M CA at a ratio of 7 parts by weight CA to 1 part by weight UW soybean hulls for 2.5 hr in order for the hulls to become saturated with CA. The hulls are collected onto a dewatering screen to remove excess (unimbibed) CA where an estimated 5% loss of unimbibed CA and 5% loss of hull fines occur.

The CA-soaked soy hulls are modified by conveying them through a vibrating fluid bed dryer with a residence time of 1.5 hr at 120°C. After modification, the yield of hulls is estimated at 110%, due to the added weight of the reacted CA. The soybean hulls are washed with deionized water for 2 hr at 25°C in a water soak tank in order to remove unreached CA. The excess, dilute CA from the water soak tank is collected from the dewatering screen, transported to the CA recovery unit (evaporator), where the liquid is concentrated to 0.6 M in CA and recycled back to the CA storage tank for recycled acid. We estimate a recovery of 72% of the unreached acid for each modification cycle.

The modified hulls are dried in a vibrating fluid bed dryer at 50°C to a moisture content of <10%. Drying time is estimated at 2 hr. The final product is stored until it is ready for bagging and shipping. From an initial 10,000 kg of UW hulls as starting

material, we estimate that 7900 kg of CA-modified soybean hulls will constitute the final product for an overall yield of 79%.

Cost estimates for the production of citric acid-modified soybean hulls were developed for the optimized process. Because the process was scaled up linearly from laboratory quantities, the scale-up may possess errors in amounts of materials needed to produce the CA-modified soybean hulls. The following considerations were excluded from the cost analysis study: working capital, marketing and distribution expenses, income and property taxes, and the cost of capital. Cost estimates were based on a feed rate of 10,000 kg/day of soybean hulls and a soybean hull yield of 79%. Therefore, the daily output of modified soybean hulls would be approximately 7900 kg/day. This estimate was made with the assumption that 72% of the original, virgin citric acid could be recovered from the water wash unit, concentrated by evaporation and recycled back to the process at a concentration of 0.6 M. Operating expenses were estimated based on the cost of raw materials, utilities, operating and maintenance labor and supplies, facility overhead charges and depreciation on the equipment costs. The final cost to modify the soybean hulls by this process was determined to be approximately $1.17/kg.

Summary

A modification process was developed in which soybean hulls were modified with CA to enhance the hulls ability to adsorb metal ions. This process yielded modified hulls with excellent adsorption characteristics similar to commercial chelating and cation exchange resins and toward a variety of metal ions. The modified hulls proved quite durable compared to commercial resins except at elevated temperatures. These positive results prompted us to reduce process steps in order to produce a competitively priced product. Based on a process flow diagram, equipment, capital and production cost estimates were made. We have completed the first steps toward transferring our technology to interested and motivated parties who seek value-added products from agricultural by-products.

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Table 1. Adsorption capacities and total negative charge of unwashed (UW), base-washed (BW) and BW, CA-modified hulls.

Sample	Adsorption capacity[1] (mmoles Cu^{2+}/g)	Total negative charge[1,2] (mmoles H^{+}/g)
UW hulls (Control)	0.32	1.33
BW hulls	0.39	1.01
BW + 0.1 M CA	0.68	1.16
BW + 0.3 M CA	1.20	2.78
BW + 0.6 M CA	1.76	4.23
BW + 0.9 M CA	2.13	4.74
BW +1.2 M CA	2.44	5.22

[1]Values are means of duplicate samples.
[2]Total negative charge determined at pH $\leq$ 3.0.

Table 2. Metals adsorption[1] for BE, CA-modified soybean hulls and commercial chelating and cation exchange resins.

Adsorbent	mmoles of metal adsorbed/g adsorbent[2]										
	Cd^{2+}		Cu^{2+}		Ni^{2+}		Pb^{2+}		Zn^{2+}		Total
Modified soy hulls	0.20	**0.67**	0.73	**0.72**	0.12	**0.62**	0.58	**0.77**	0.12	**0.62**	1.74
Amberlite IRC-718	0.00	**0.90**	1.13	**0.94**	0.10	**0.96**	0.08	**0.91**	0.02	**0.99**	1.33
Duolite GT-73	0.03	**0.61**	0.66	**0.62**	0.00	**0.12**	0.18	**0.46**	0.01	**0.54**	0.88
Amberlite IR-122	0.28	**0.65**	0.14	**0.54**	0.30	**0.62**	0.41	**0.61**	0.29	**0.74**	1.42
Amberlite 200	0.24	**0.62**	0.09	**0.50**	0.18	**0.54**	0.54	**0.68**	0.19	**0.69**	1.24

[1]Two solutions were used. One solution contained 7 mM concentration of each metal (total metals concentration of 35 mM) in buffer at pH 4.8. The other solution contained 7 mM concentration of a single metal in buffer. Adsorption values in boldface indicate adsorption from a solution containing 7 mM concentration of a single metal.
[2]All values are means of duplicate determinations.

Table 3a. Attrition of BW, CA-modified soybean hulls and two commercial ion exchange resins[1].

Sample Identification	Attrition (%)[2]
Modified hulls	7.0
IRC-718	18.8
IR-122	12.0

[1]Samples were stirred at 25°C and in pH 4.8 buffer with a Teflon stir bar at 300 rpm for 24 hr.
[2]Mean of duplicate determinations.

Table 3b. Attrition of BW, CA-modified soybean hulls and two commercial ion exchange resins at different exposure temperatures[1].

Sample Identification	Attrition (%)[2]			
	25°C	45°C	65°C	85°C
Modified hulls	0.6	3.9	18.8	33.1
IRC-718	9.2	8.0	9.4	8.2
IR-122	<0.1	<0.1	<0.1	<0.1

[1]Samples were shaken at 300 rpm in an orbital shaker bath in pH 4.8 buffer for 24 hr.
[2]Mean of duplicate determinations.

Table 3c. Attrition of BW, CA-modified soybean hulls and two commercial ion exchange resins at different pH values[1].

Sample Identification	Attrition (%)[2]		
	pH = 3.0	pH = 7.0	pH = 11.0
Modified hulls	4.9	10.4	12.0
IRC-718	13.9	21.9	7.4
IR-122	3.4	0.4	<0.1

[1]Samples were stirred at 300 rpm with a Teflon stir bar at 25°C for 24 hr.
[2]Mean of duplicate determinations.

Table 4. Adsorption capacities and total negative charge for base washed (BW), unwashed (UW) and water washed (WW), citric acid modified soybean hulls.

Sample	Adsorption capacity[1] (mmole Cu^{2+}/g)	Total negative charge[1,2] (mmoles H^{+}/g)
BW	1.70	4.61
UW	1.86	4.60
WW	1.93	4.72

[1]Means of duplicate samples.
[2]Total negative charge determined at pH $\leq$ 3.0.

Table 5. Comparison of copper ion adsorption[1] and reaction efficiency for UW, CA-modified soybean hulls with and without a drying step prior to modification.

Sample	Copper ion adsorption[2] (mmoles Cu^{2+}/g)	Reaction Efficiency[2] (%)
UW-A (2 hr at 50°C, then 1.5 hr at 120°C)	1.70	41.7
UW-B (1.5 h at 120°C)	1.60	27.1
UW-C (2 h at 120°C)	1.65	41.2
UW-D (3 h at 120°C)	1.73	54.0

[1]The assay solution contained 20 mM $CuCl_2$ in buffer at pH 4.8.
[2] Means of duplicate samples.

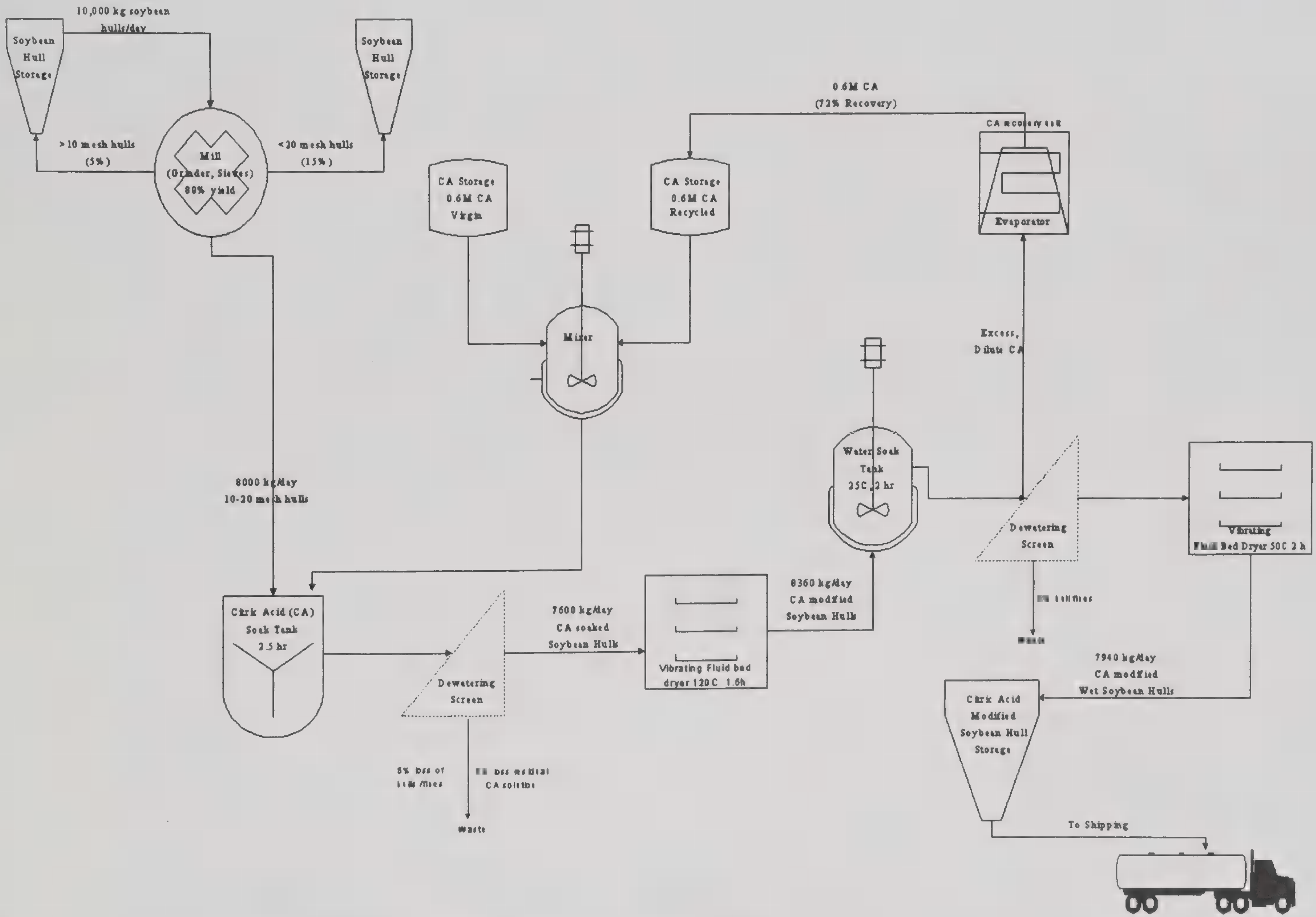

Figure 1. Process flow diagram for CA modification of soybean hulls.

50th Oilseed Conference

Tools for Improving Profits

January 21-23, 2001 ✹ Hyatt Regency New Orleans

Effects of Oilseed (Peanuts) on Appetite, Food Intake, Energy Balance, and Cardiovascular Risk

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Effects of Oilseed (Peanuts) on Appetite, Food Intake, Energy Balance and Cardiovascular Risk

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Important Nutritional Characteristics of Nuts

energy (160-200kcal/oz)
protein (10-25% by weight - high arginine)
fiber (5-11% by weight)
polyunsaturated fat/monounsaturated fatty acids
micronutrients (e.g., magnesium, copper, zinc, potassium, folic acid, vitamins E & B_6)
phytochemicals (e.g., isoflavones, flavonoids, phenolic compounds, resveratrol)

Role of Fats in Cardiovascular Risk Reduction

Katan Am J Clin Nutr 1997;66(Supple):974S
- Meta-analysis (N=682)
  - Low-fat diets reduce plasma cholesterol - LDL AND HDL
  - HDL declined 0.5mg/dl per 1% energy shift from SFA to CHO
  - Substitution of SFA with CHO - no change of HDL/LDL ratio
  - Substitution of SFA with MUFA - higher HDL/LDL ratio

Effects of Nut Consumption on Cardiovascular Risk

Epidemiological Studies

Adventist Health Study (N=34,198) (Fraser et al., Arch Int Med 1992;152:1416)
- Consumption of nuts 1-4X/wk vs <1X/wk
  - 22% lower risk of non-fatal, acute myocardial infarction
- Consumption of nuts $\geq$5X/wk vs <1X/wk
  - 51% lower risk of non-fatal, acute myocardial infarction
- Benefits occurred independent of age, gender, body weight, smoking status, physical activity, blood pressure

Iowa Women's Health Study (N=34,500) (Kushi et al., NEJM 1996;334:1156)
- Consumption of nuts >1X/wk vs <1X/wk

40% reduction of Coronary Heart Disease

Nurses Health Study (N=86,016) (Hu et al., BMJ 1998;317:1341)
Consumption of nuts ≥5X/month vs <1X/month
35% lower risk of coronary heart disease risk
Benefits occurred independent of body mass index, smoking status, physical activity, alcohol use, multivitamin use, or fruit and vegetable intake

Intervention Studies

Sabate et al., NEJM 1993;328:603.
Randomized, cross-over study - low fat with or without walnuts (20% of energy)
Reduced total Cholesterol - 12%
Reduced low density cholesterol - 15%

O'Bryne et al., (N=25 hypercholesterolemic females - 6 months) Lipids 1997;32:687.
Parallel Group Study - low fat MUFA diet (26% of fat) (high oleic peanuts)
Reduced total cholesterol 10%
Reduced LDL cholesterol 12%

Spiller et al. (N=45 hyperlipidemic men and women - 4 weeks) J Am Col Nutr 1998;17:285
Parallel group Study
Almond diet (100g/d - 53% MUFA, 13% PUFA, 10% SFA)
Olive oil diet (48g/d + 113g/d cottage cheese - 65%PUFA, 8%PUFA, 14%SFA)
Dairy diet (85g/d cheddar cheese + 28g/d butter - 31%MUFA, 6%PUFA, 37%SFA)
Almond diet led to greater reduction of total cholesterol, LDL cholesterol and total cholesterol: HDL cholesterol ratio
Olive oil diet - no changes
Dairy diet - total cholesterol, LDL cholesterol increased

Pearson et al. (N=22 - 25 days) (EB, 1998)
Cross-over Study
Average American Diet (35% fat, 15% SFA)
Low Fat (25% fat, 7% SFA)
Olive Oil (35% fat, 6% saturated)
Peanut/Peanut Butter (35% fat, 7% SFA)
Peanut Oil (35% fat, 7% SFA)
All diets reduced total cholesterol 9-12%
All diets reduced low density cholesterol 12-16%
*Low fat diet raised triglycerides, others lowered

Hyson et al., (N=16 - 6 weeks) (EB 1998)

Cross-over Study

Replaced 50% of fat intake with almonds or almond oil

Both diets reduced plasma LDL cholesterol 11-12%

Curb et al., (N=30 - 30 days) (EB 1998)

Cross-over Study

Macadamia nuts - 37% kcal from fat, 9% saturated

AHA step 1 diet - 30% kcal from fat, 9% saturated

Typical U.S. diet - 37% kcal from fat, 15% saturated

cholesterol reduced by 5%, LDL Cholesterl reduced by 7.5% and HDL cholesterol reduced by 3.6% on Macadamia nut diet relative to typical diet

Triglycerides were 10% lower on Macadamia diet than AHA diet

Zambon et al. (N=49 hyperlipidemic subjects) Ann Intern Med 2000;132:538.

Randomized, cross-over study - low fat with or without walnuts

Olive Oil Diet (30% kcal from fat, 21% MUFA, 4% PUFA, 5%SFA)

Walnut Diet (33% kcal from fat, 16% MUFA, 12% PUFA, 5%SFA)

Walnut Diet reduced total Cholesterol, LDL cholesterol and apolipoprotein(a) - 4-6% compared to the olive oil diet

Iwamoto et al., (N=40 - 4 weeks) J Nutr 2000;130:171.

Cross-over Study - traditional Japanese diet with (12.5% kcal) or without walnuts

Total cholesterol decreased 4% (men) and 5% (women)

LDL cholesterol decreased 9% (men) and 11% (women)

Lermer & Mattes (N=15 normolipedemic subjects) - NEW DATA

Cross-over Study

Free-feeding - 8 weeks with 500kcal peanuts/peanut butter

Substitution - 8 weeks with 500kcal peanuts/peanut butter

Supplementation - 3 weeks with 500kcal peanuts/peanut butter

Significant reduction of triglycerides during free-feeding

Effects of Nut Consumption on Appetite and Body Weight

Epidemiological Studies

Hu et al. (See above)

Inverse association between frequency of nut consumption and body mass index

Fraser et al. (see above)

Inverse association between frequency of nut consumption and body mass index

Intervention Studies

O'Bryne et al., (see above)

Significant reduction of body weight and BMI while on a low fat but high MUFA diet compared to just a low fat diet

Spiller et al (see above)

Significant elevation of energy intake required to maintain body weight while adhering to a

high MUFA diet

Kirkmeyer & Mattes (N=24) Int'l J Obes 2000;24:1167

Preload Study (peanuts, peanut butter, almonds, chestnuts, chocolate, rice cakes, pickles, no load)

Energy-matched nut preloads reduced hunger comparably and more than weight or volume controls

Dietary energy compensation for nut preloads was precise

Lokko et al. (N=26) - NEW DATA

Preload Study (peanuts, peanut butter, almonds, chestnuts, chocolate, rice cakes, pickles, no load)

Energy-matched nut preloads reduced hunger comparably and more than weight or volume controls

Dietary energy compensation for nut preloads was precise

Lermer & Mattes (N=15) - NEW DATA

Cross-over Study

Free-feeding - 8 weeks with 500kcal peanuts/peanut butter

Substitution - 8 weeks with 500kcal peanuts/peanut butter

Supplementation - 3 weeks with 500kcal peanuts/peanut butter

No significant change of appetite during any treatment

No significant change of body weight during free-feeding

Alfenas & Mattes (N=20) - NEW DATA

Preload Study (muffins with peanut oil, canola oil, butter or no fat)

50*th*
Oilseed
Conference

Tools for Improving Profits

January 21–23, 2001 ✵ Hyatt Regency New Orleans

Project Specific Web Sites

Grant Mitchell
Process Plus, Cincinnati, OH

Project Specific Web Sites

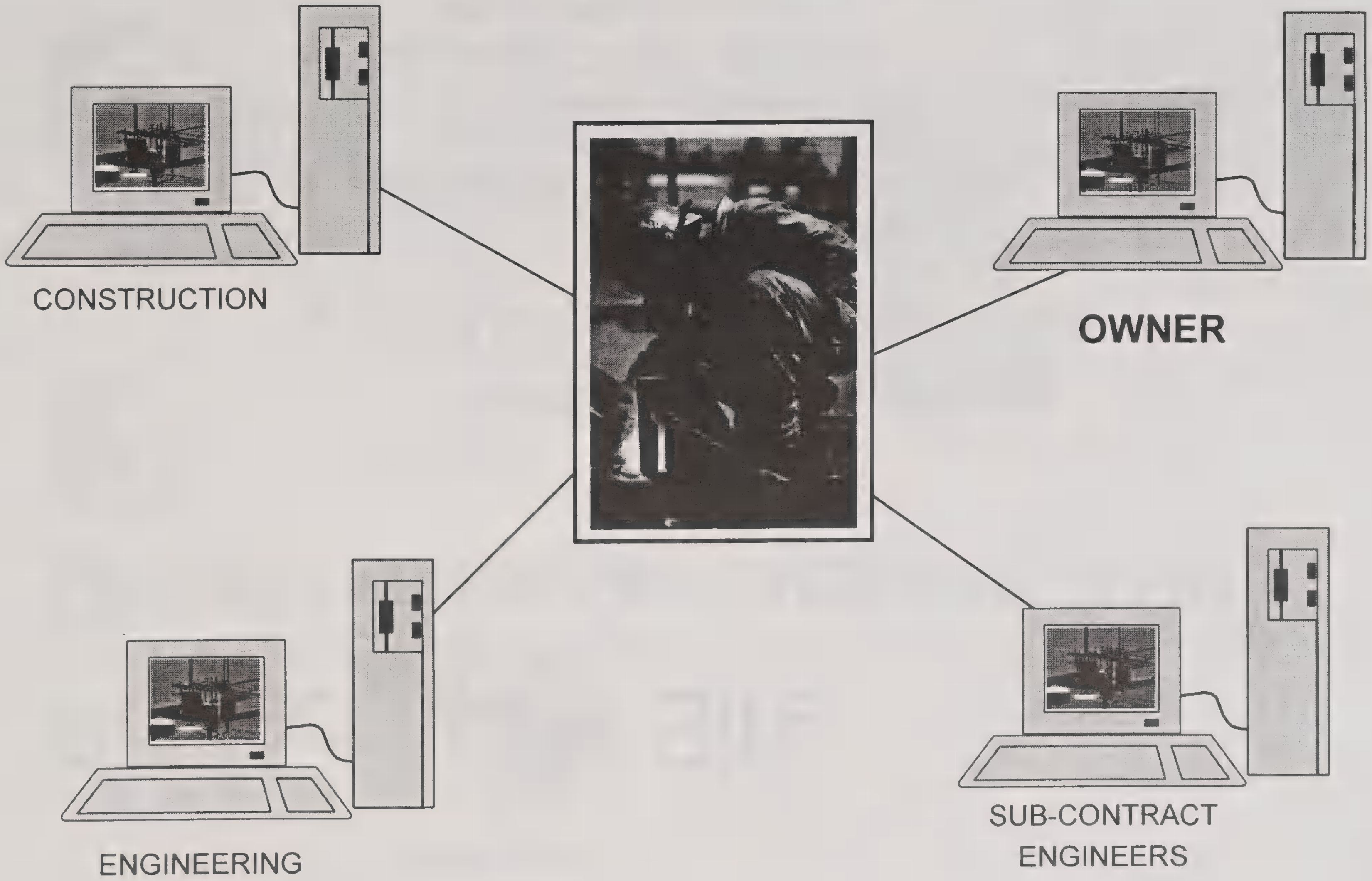
A Web based tool enabling companies to manage project life cycles utilizing multiple contractors from concept to completion through a centralized database of project and business information

Project Web Site

Project Data Access via the World Wide Web

Project Web Site

Project Data Access via the World Wide Web



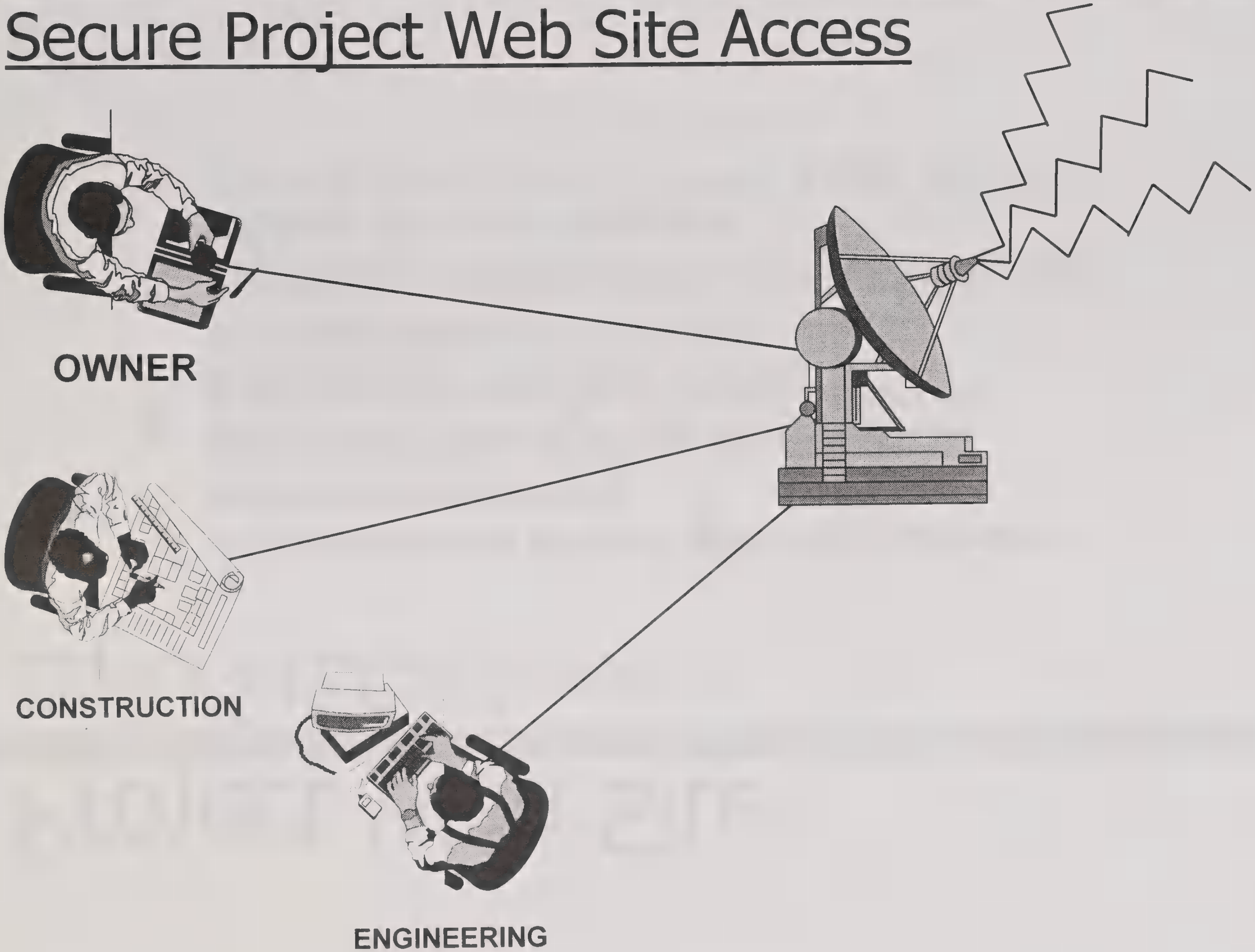
Project Web Site

Complete Project Communication System

- **SECURITY**
  - Password protected
  - Access controlled
- **CENTRALIZED INFORMATION REPOSITORY**
  - Links to outside data
  - Drag-and-drop document addition
- **AUTOMATIC CHANGE NOTIFICATION**
  - Team members “subscribe” to site data
  - System makes notification of changes
- **ONLINE DOCUMENT REVIEWS**
  - Browser-based file viewers
  - Team members post comments, mark up documents, or attach files
- **INFORMATION REQUEST TRACKING**
  - Team members post requests
  - System routes request through E-Mail
  - Dedicated request logs

Project Web Site

Secure Project Web Site Access



Project Web Site

Third Party Collaboration

- **All trades on a project communicate and share data instantly**
- **Real-time message tracking reduces delays and enhances project accountability**
- **Increases productivity while reducing the chance for legal disputes**

Project Web Site

Accessibility

- **View project data 24 hours a day 365 days a year**
- **Access the system from any computer with current Internet browsers**
- **Project information is never removed - report on past projects for valuable historical data**

Project Web Site

Centralized Database

- **More than a single project Web site, the system handles every project the enterprise bids**
- **Report across multiple projects to get a "Big Picture" view of company activities**
- **Store thousands of individual companies and employees, and easily assign them to new or existing projects - never search for phone numbers again**

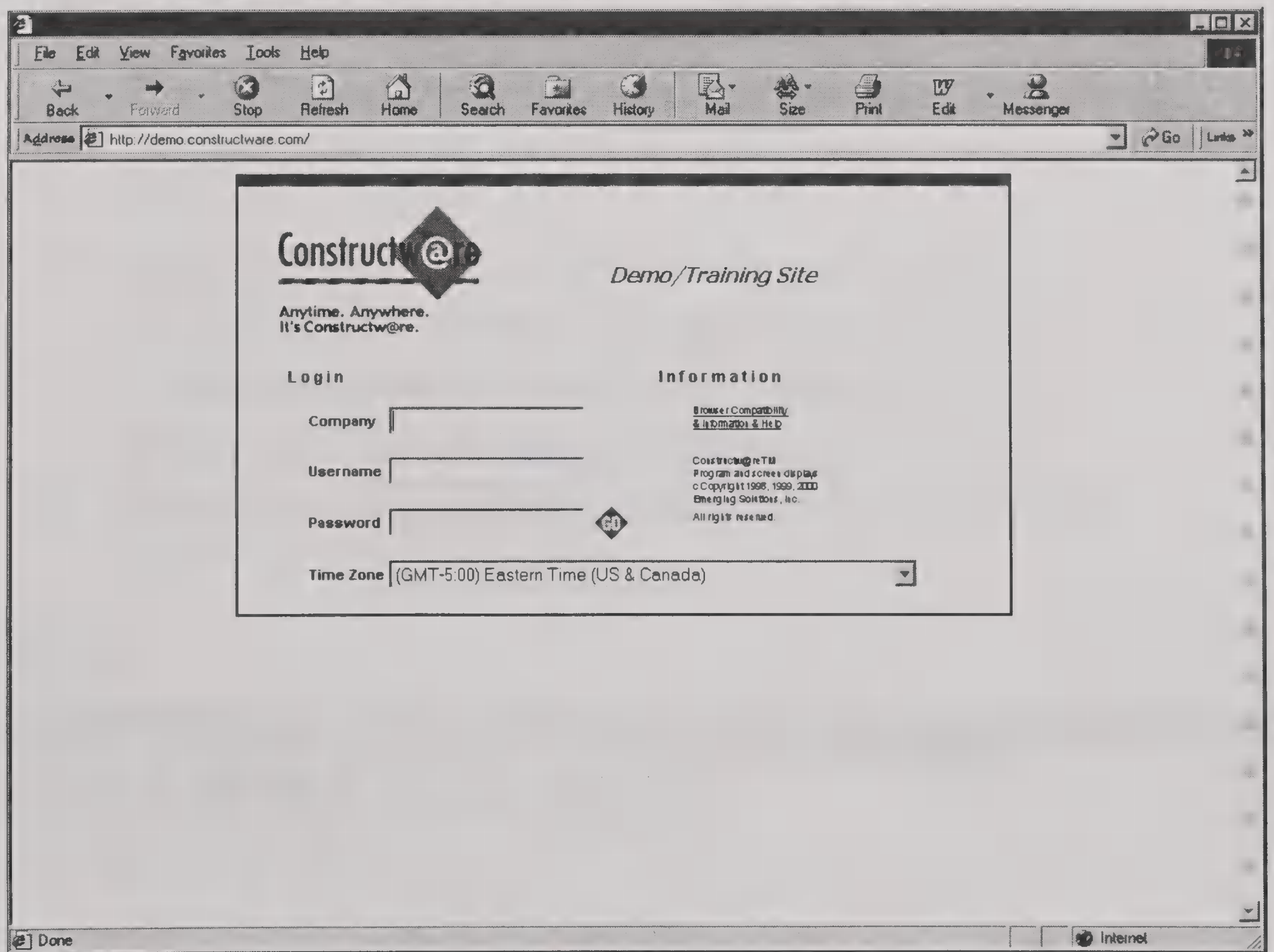
Project Web Site

Security

- **Each user is assigned a unique, three-step password to access the system**
- **Users have pre-defined security levels, limiting their access throughout the application**

Project Web Site

Secure Project Web Site Access



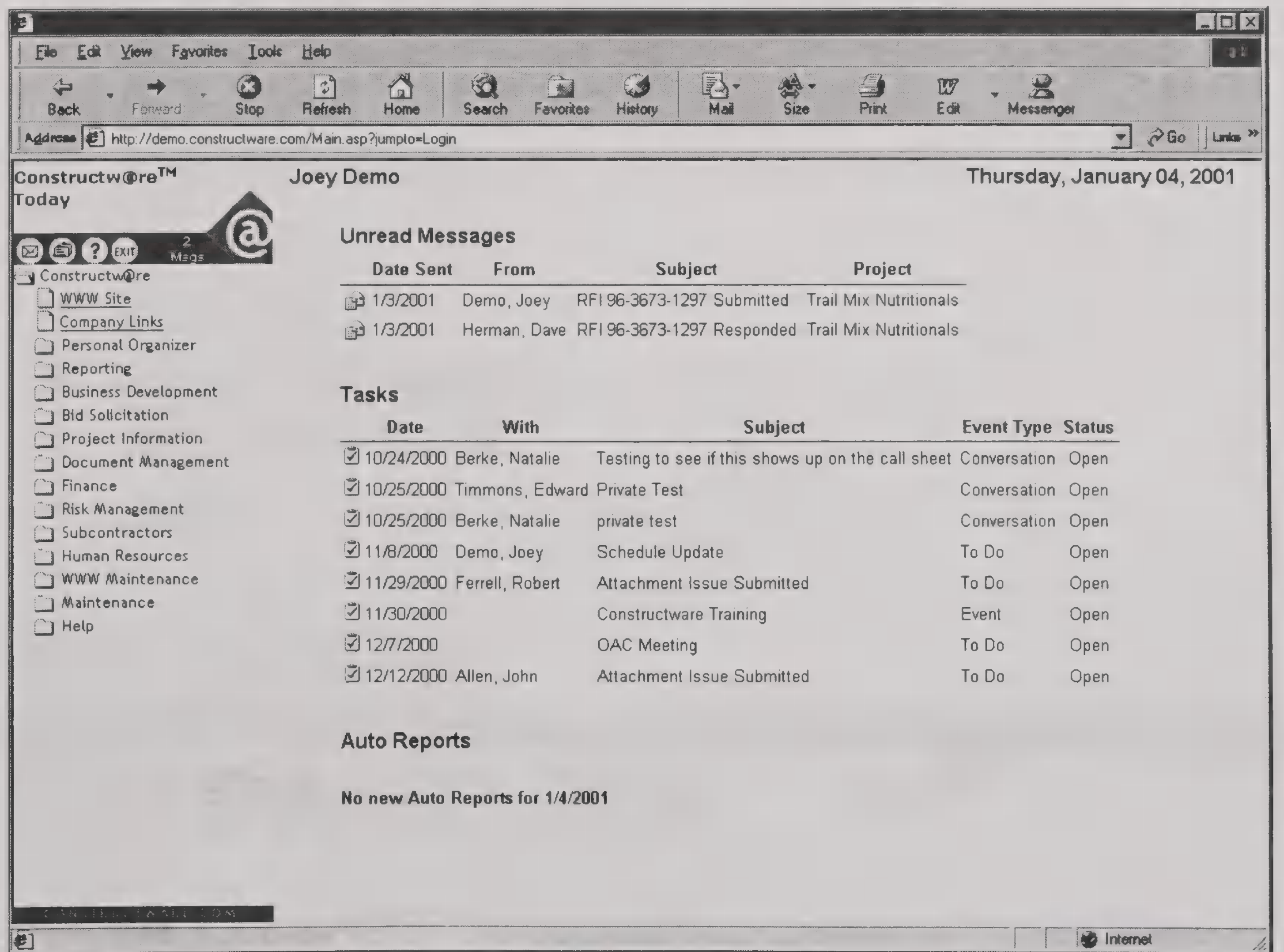
Project Web Site

Personal Organizer

- **Today**
- **Messaging Inbox**
- **Messaging Outbox**
- **Task List**
- **Call Log**
- **Change Password**
- **Dashboard**
- **Send E-Mail**
- **Vacation Info**

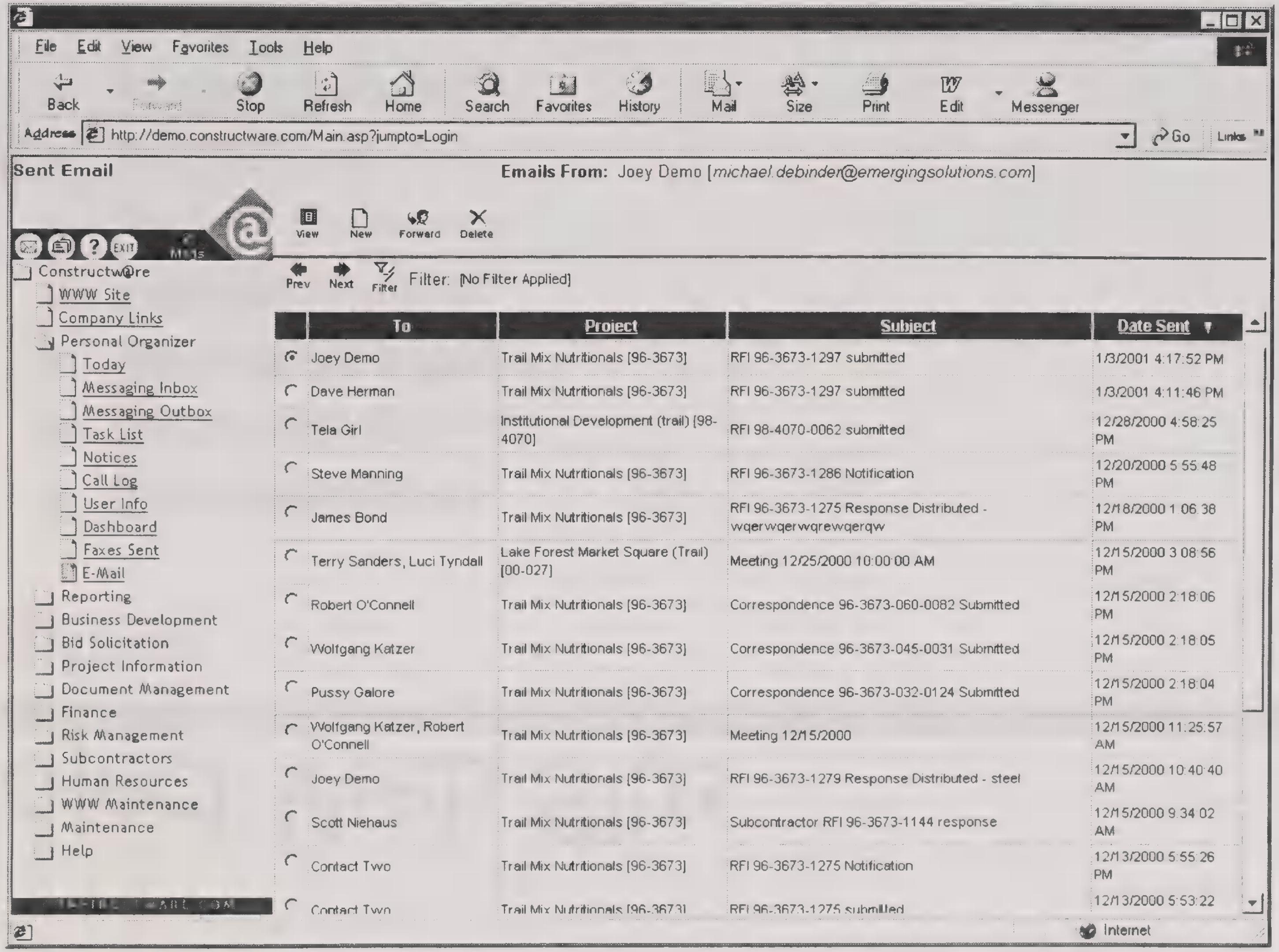
Project Web Site

Personal Entry Page



Project Web Site

E-mail



Project Web Site

Reporting

- **General Reports**
- **Auto Reports**
- **Outstanding Events**
- **Infodex**

Project Web Site

Business Development

- **Project Details**
- **Projections**
- **Project Summary**
- **Prebid Checklist**
- **Project Proposal**
- **Projection Reports**
- **Competition**

Project Web Site

Project Information

- Project Defaults
- Contract/Bond
- Contract History
- Team Members
- Phones
- Project CSI Codes
- Assign Companies
- Discussion Forum

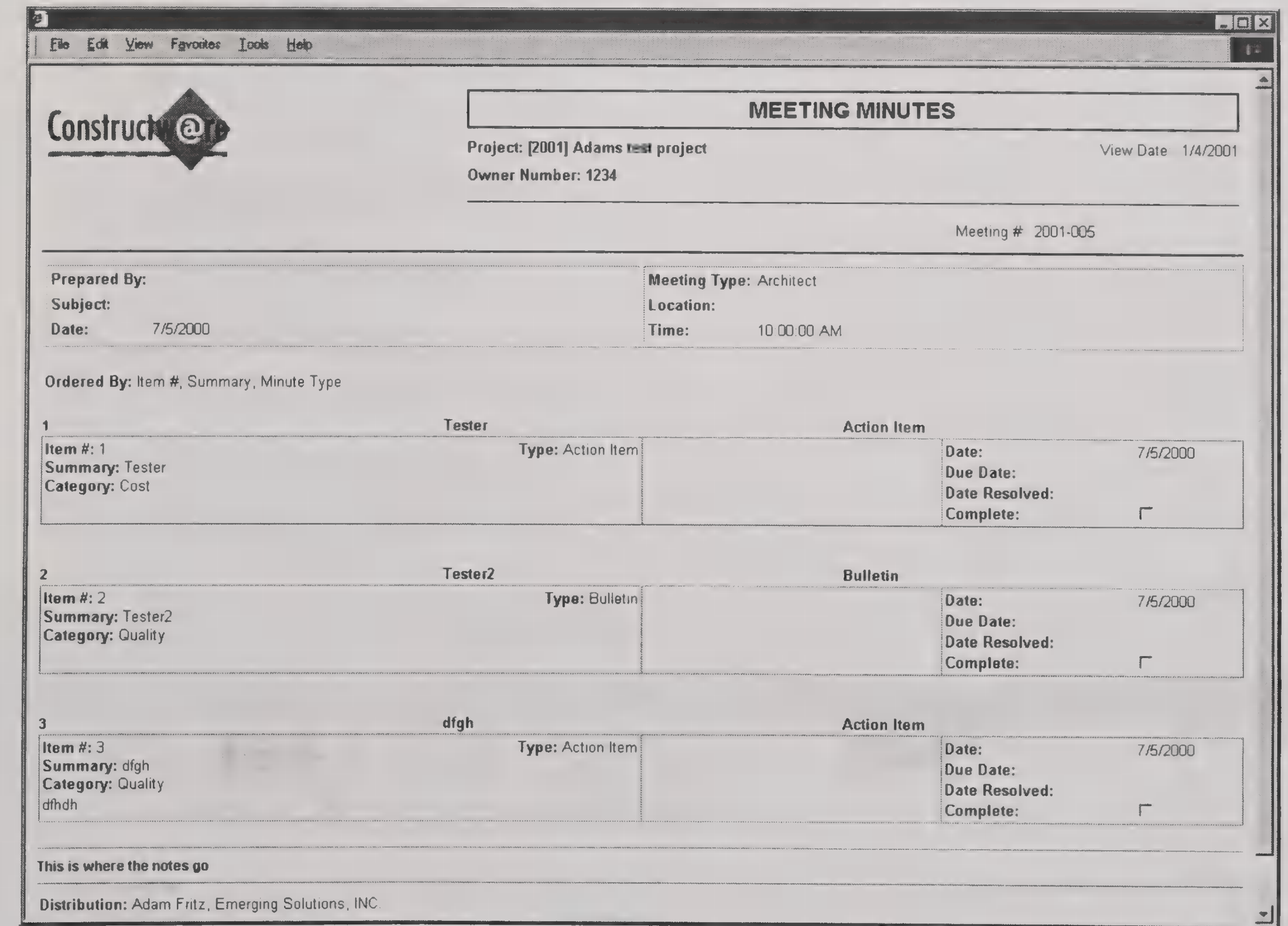
Project Web Site

Document Management

- **Attachments**
- **Correspondence**
- **Custom Forms**
- **Daily Reports**
- **Design Review**
- **Distribution**
- **Documents**
- **Drawing Log**
- **Meetings**
- **Owners**
- **Punch Lists**
- **RFI's**
- **Submittals**
- **Transmittals**

Project Web Site

Meeting Minutes



File Edit View Favorites Tools Help

Construct@re

MEETING MINUTES

Project: [2001] Adams [redacted] project View Date 1/4/2001

Owner Number: 1234

Meeting # 2001-005

Prepared By:	**Meeting Type:** Architect
Subject:	**Location:**
Date: 7/5/2000	**Time:** 10:00:00 AM

Ordered By: Item #, Summary, Minute Type

1 **Tester** **Action Item**

Item #: 1 **Summary:** Tester **Category:** Cost	**Type:** Action Item	**Date:** 7/5/2000 **Due Date:** **Date Resolved:** **Complete:**

2 **Tester2** **Bulletin**

Item #: 2 **Summary:** Tester2 **Category:** Quality	**Type:** Bulletin	**Date:** 7/5/2000 **Due Date:** **Date Resolved:** **Complete:**

3 **dfgh** **Action Item**

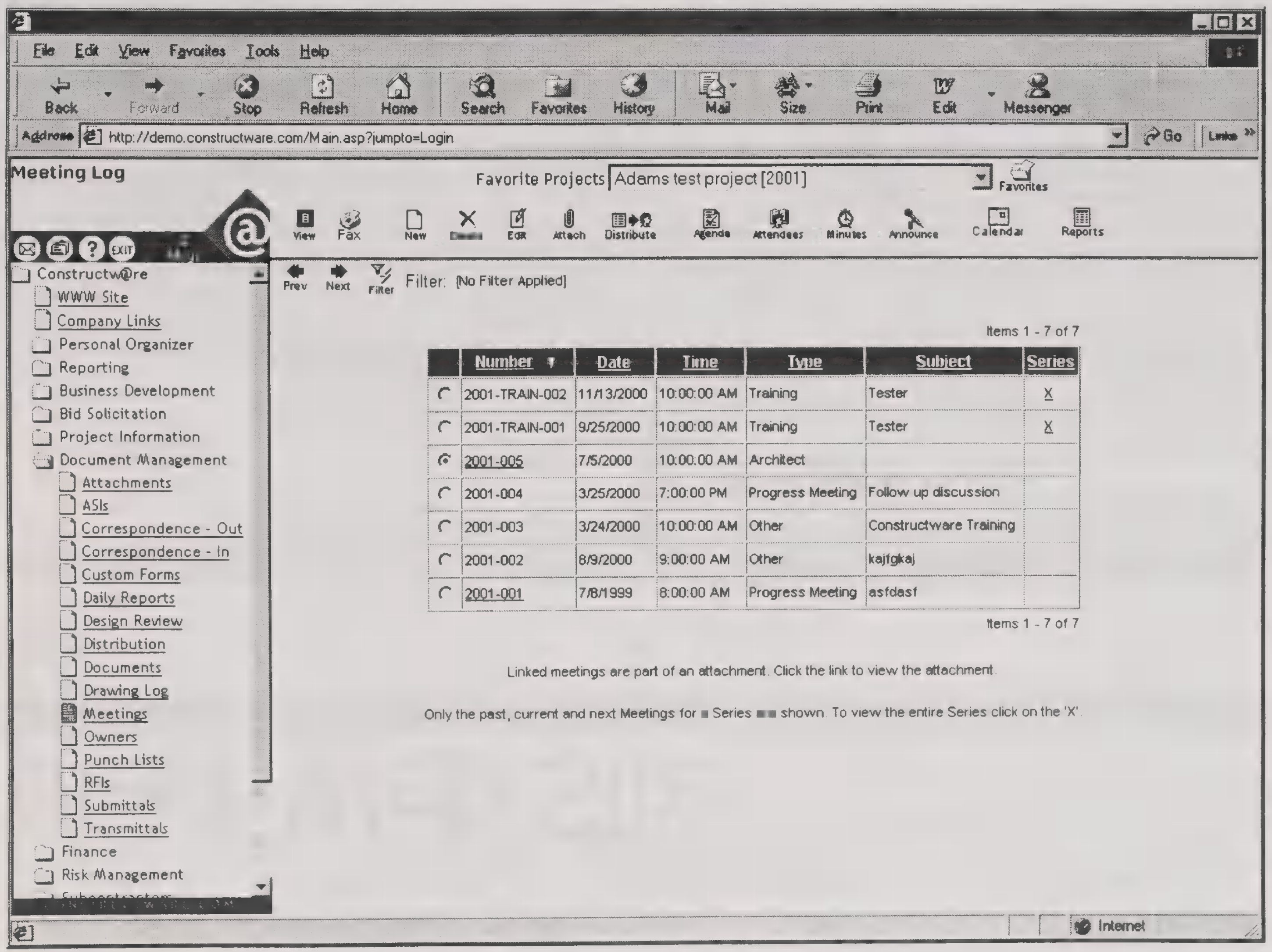
Item #: 3 **Summary:** dfgh **Category:** Quality dfhdh	**Type:** Action Item	**Date:** 7/5/2000 **Due Date:** **Date Resolved:** **Complete:**

This is where the notes go

Distribution: Adam Fritz, Emerging Solutions, INC.

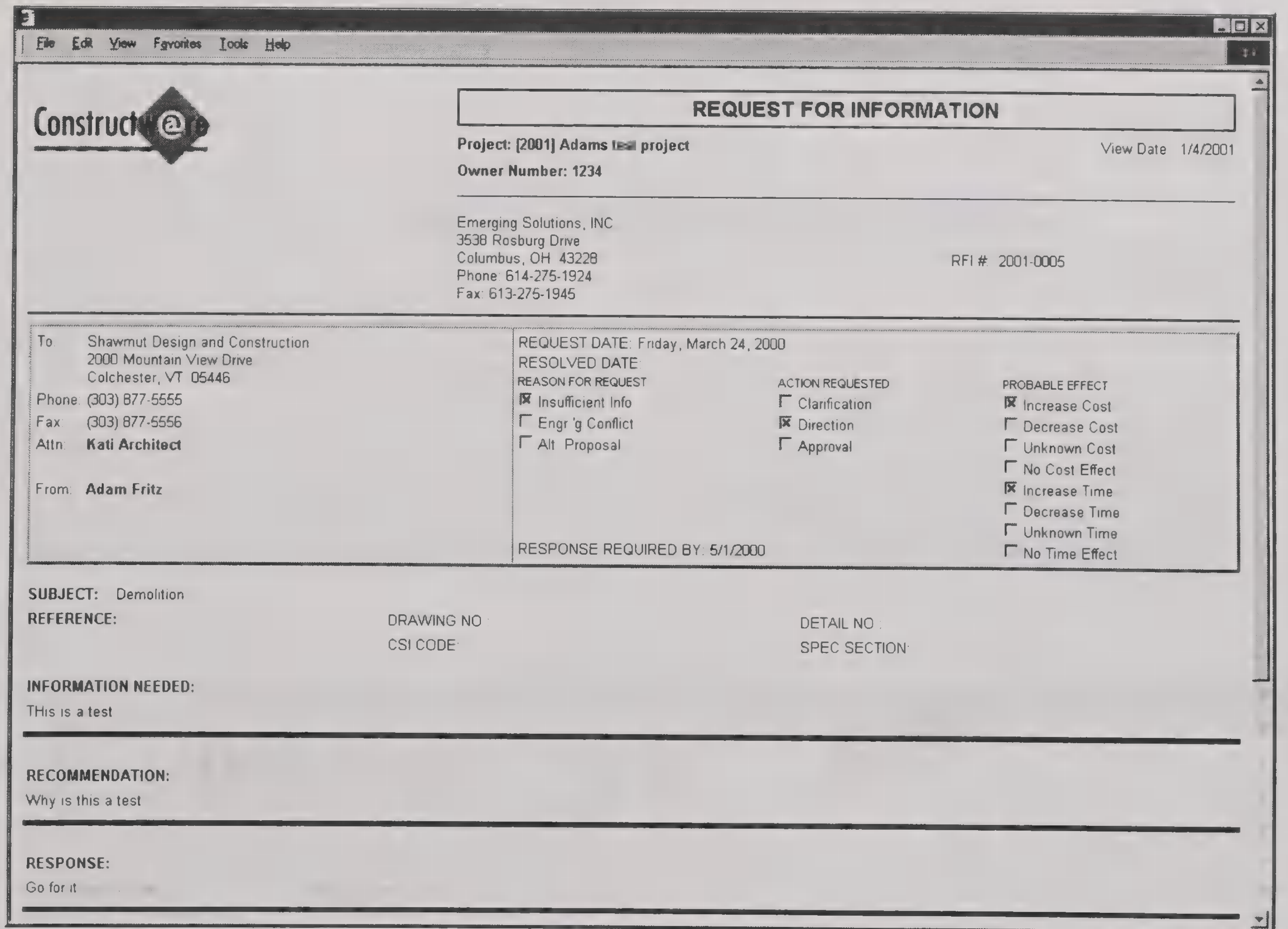
Project Web Site

Meeting Log



Project Web Site

RFI's



File Edit View Favorites Tools Help

REQUEST FOR INFORMATION

Project: [2001] Adams [redacted] project View Date 1/4/2001

Owner Number: 1234

Emerging Solutions, INC
3538 Rosburg Drive
Columbus, OH 43228
Phone 614-275-1924
Fax: 613-275-1945

RFI # 2001-0005

To Shawmut Design and Construction 2000 Mountain View Drive Colchester, VT 05446 Phone (303) 877-5555 Fax (303) 877-5556 Attn **Kati Architect** From **Adam Fritz**	REQUEST DATE: Friday, March 24, 2000 RESOLVED DATE REASON FOR REQUEST: ☒ Insufficient Info ☐ Engr'g Conflict ☐ Alt Proposal ACTION REQUESTED: ☐ Clarification ☒ Direction ☐ Approval PROBABLE EFFECT: ☒ Increase Cost ☐ Decrease Cost ☐ Unknown Cost ☐ No Cost Effect ☒ Increase Time ☐ Decrease Time ☐ Unknown Time ☐ No Time Effect RESPONSE REQUIRED BY: 5/1/2000

SUBJECT: Demolition

REFERENCE: DRAWING NO CSI CODE DETAIL NO SPEC SECTION

INFORMATION NEEDED:

THis is a test

RECOMMENDATION:

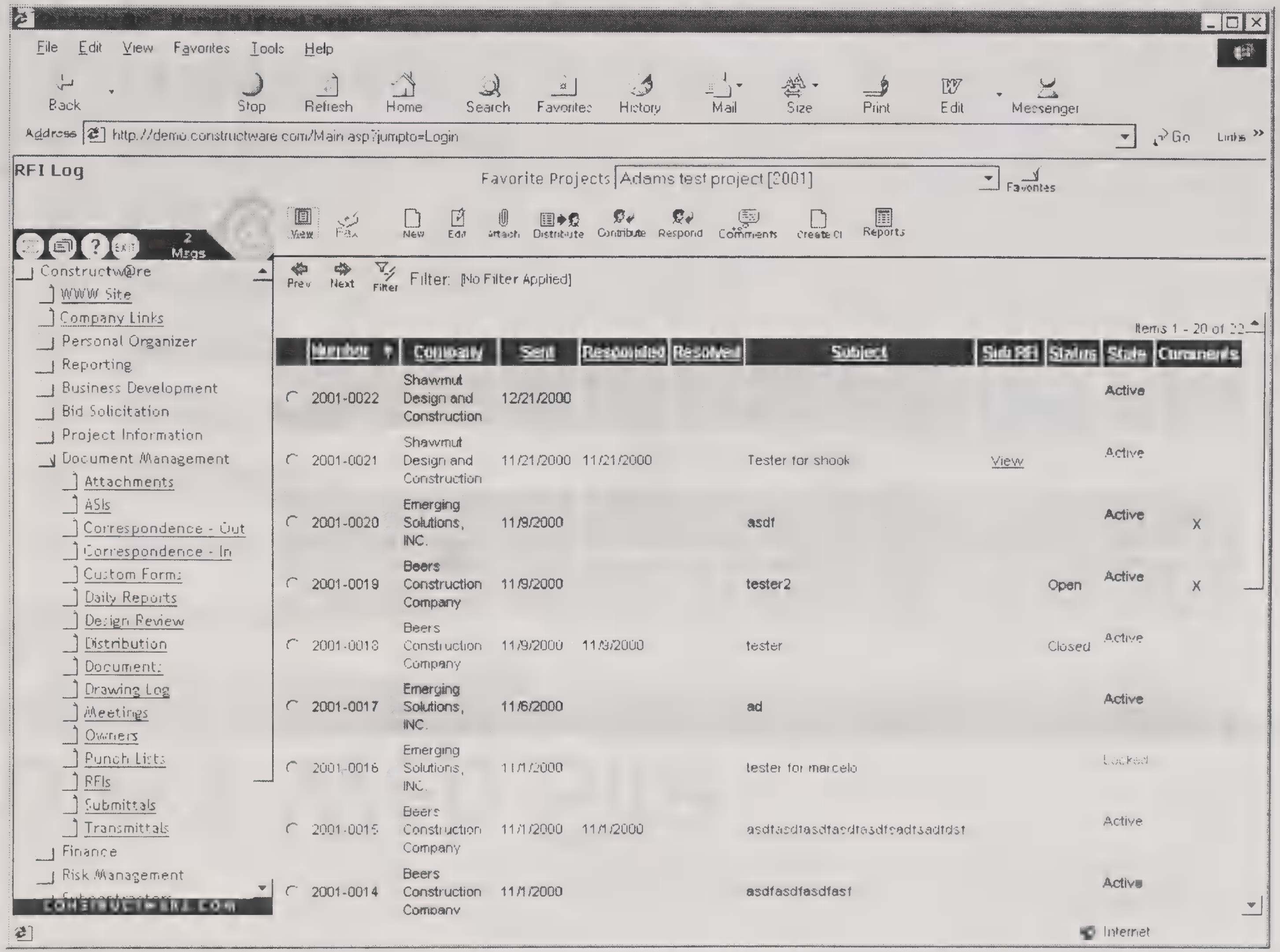
Why is this a test

RESPONSE:

Go for it

Project Web Site

RFI Log



Project Web Site

Finance

- **Bonds**
- **Budgets**
- **Insurance**
- **Insurance Defaults**
- **Purchase Orders**
- **POCO's**
- **PayApps**
- **Change Items**
- **Request CO's**
- **Owner CO's**
- **Sub CO's**
- **SOV Setup**
- **Sub Change Req**
- **Subcontracts**

Project Web Site

Human Resources

- **Employee Info**
- **Confidential Info**
- **Benefits**
- **Wages**
- **Dependents**
- **Education**
- **Evaluation**
- **Vacation/Comp**
- **Batch Updates**
- **Assign Companies**

Web Based Information Site

A centralized, real-time database fully accessible over the Internet, improving communication and accountability across multiple projects and company locations

Q: Why did Federal Express revolutionize the package delivery business?

Better employees than UPS?

Better trucks than the Post Office?

Better packaging?

Better rates?

ANSWER:

Better information system:

- customers knew where package was
  - results were guaranteed
    - "absolutely, positively"

The FUTURE:

UNIVERSAL ACCESS TO KEY INFORMATION

- CPM Schedule
- Photos
- Drawings
- RFI's
- Meeting Minutes
- Change Orders

SOLUTION

The Internet

Project Web Sites

....a better way to communicate on your next successful project

50th
Oilseed
Conference

Tools for Improving Profits

January 21-23, 2001 ✷ Hyatt Regency New Orleans

Internet Tools for Improving Profits

Robert L. Stroup
The Robert L. Stroup Company Ltd.,
Troy, OH

2001 OILSEED CONFERENCE

Internet Tools

------*for improving Profits*

by

Robert L. Stroup

The R.L. Stroup Company Ltd.

INTERNET TOOLS

Handy Search Engines and Websites for Oilseed People

Web Pages

2,000,000,000

Two Billion
Indexable Pages*

*PC Magazine December 5, 2000

INTERNET TOOLS

Search Engines

Websites

Search Engines

The BIG SIX-1999

- Alta Vista
- Excite
- HotBot
- Infoseek
- Lycos
- Yahoo

Search Engines

The BIG SIX-2001

- Direct Hit
- Google
- HotBot
- MSN
- Northern Light
- Oingo

Ask Jeeves

An Ask Jeeves Service

More accurate results on the first try!

direct hit

One Search Engine. Millions of Minds.

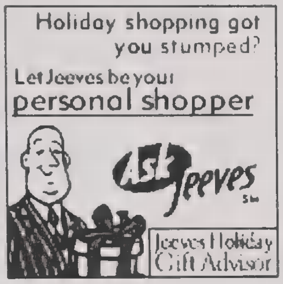
Search for...

web sites

Advanced Search

Tip: word order does not matter

January 4, 2001



direct hit search results are also featured on

WebWatch

Check out the most popular web sites for these frequently searched terms:

Pokemon
Napster
The WWF
Martha Stewart
Auctions
Personals
Games

Arts
Television, Movies, Music

Business
Employment, Industries, Investing

Computers
Internet, Software, Hardware

Games
Video Games, RPGs, Gambling

Health
Fitness, Medicine, Alternative

Home
Kids, Homeowners, Cooking

News
Weather, Media, Newspapers

Recreation
Travel, Food, Outdoors, Humor

Reference
Maps, Education, Libraries

Regional
Countries, Asia, Europe

Science
Biology, Psychology, Physics

Society
People, Politics, Issues

Sports
Baseball, Soccer, Basketball

World
Deutsch, Español, Indonesia

Ask Jeeves **Have a Question? Ask Jeeves!**

Ask

Example: Am I in love?

Google™

Search 1,326,920,000 web pages

Advanced Search
Preferences

Google Search | I'm Feeling Lucky

Google Web Directory
the web organized by topic

Cool Jobs - Add Google to Your Site - Advertise with Us - Google in your Language - **All About Google**

LYCOSNETWORK

Lycos Home | Site Map | My Lycos

"Yeah, feeling healthy is a good thing."

Search Smarter

SEARCH

Look for:
all the words

Date:
anytime

Language:
any language

Pages Must Include:
image
MP3
video
Javascript

Return Results:
10
full descriptions

PERSONALIZE THESE SETTINGS

ADVANCED SEARCH

SUBMIT WEB SITE | HELP

Search Resources

Music Search
Free Downloads
Research Service
Find Alumni
Road Maps
Jobs & Resumés
FTP Search
Domain Names
Public Records

Shopping Resources

Lycos SHOP
Shop By Request
Books
Hardware
Classifieds
Travel
Autos
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Merchant Match

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HOTBOT DIRECTORY

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Movies, Music, Television ...

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Investing, Jobs, Industries ...

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Hardware, Internet, Software ...

Games
Videogames, Role-Playing ...

Health
Medicine, Fitness, Alternative ...

News & Media
Online, Newspapers, Weather ...

Recreation
Food, Outdoors, Humor ...

Reference
Libraries, Education, Maps ...

Regional
United States, Europe, Asia ...

Science & Technology
Technology, Social Sciences...

Society
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Search by Category

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Oingo **Meaning-Based Search** is powered by the Oingo Ontology, a highly detailed database of over 1,000,000 words and meanings, linked by millions of relationships in a semantic network that is constantly changing to reflect the currency of everyday language. Once a query is submitted, our interpretive algorithm parses it and the output is filtered to derive the optimal result.

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Business
Jobs, Industries, Investing...

Computers
Internet, Software, Hardware...

Games
Video Games, RPGs, Gambling...

Health
Fitness, Medicine, Alternative...

Home
Kids, Consumers, Recipes...

News
Media, Newspapers, Weather...

Recreation
Travel, Food, Outdoors, Humor...

Reference
Maps, Education, Libraries...

Regional
US, Canada, UK, Europe...

Science
Biology, Psychology, Physics...

Shopping
Autos, Clothing, Gifts...

Society
People, Religion, Issues...

Sports
Baseball, Soccer, Basketball...

World
Deutsch, Español, Svenska...

MEANING-BASED SEARCH (tm)

What happened to the old Dogpile?

DOGPILE

All results, no mess.

Fetch

Local Search:

Select: The Web

Help

Doggy Daily
Track down a small business near you.

Joke of the Day

Shopping
Go Shopping

Stores
REI
Amazon.com
Gap.com
AbtElectronics.com
Ashford.com

Departments
Apparel & Accessories
Electronics
Gifts & Flowers
Toys
Sporting Goods
Holiday Gifts

Marketplace

Autos
Pay Bills Online
Discount Airfares
Jobs
Travel Tools
Dogpile Radio
Get Free Email
Local Services

Dogpile References

Favorite Fetches

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Auto Insurance
Boats
Careers
Compare Prices
Data Recovery
Digital Photos
Electronics
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Mortgage
PCS Phones
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White Pages
Classifieds
Maps
Weather
Stock Quotes
Jobs/Careers
Horoscopes
Personals
City Guides
Health Center
Gov't Listings

Web Directory

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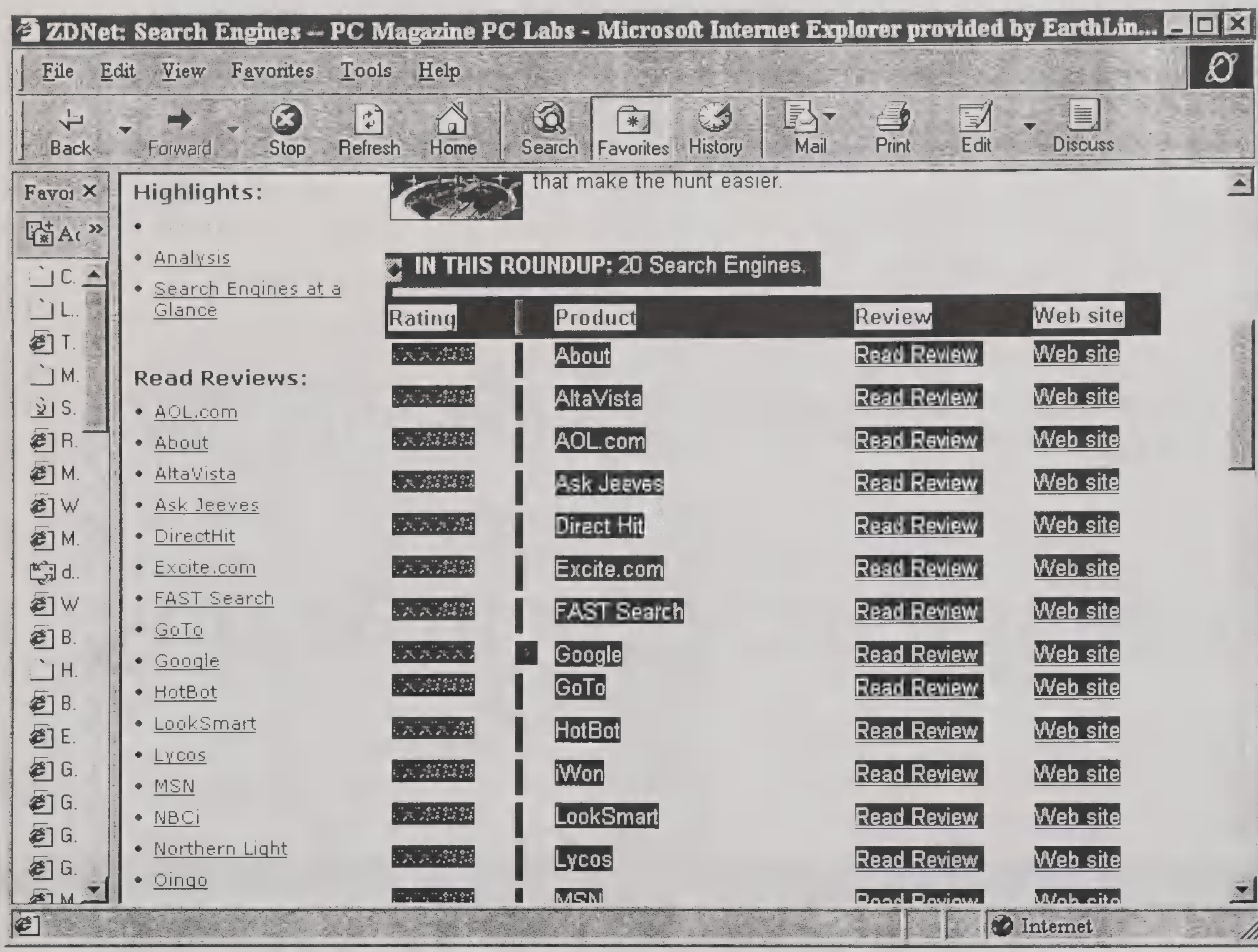
Search Engines

Survey of 20 Search Engines

PC Magazine December 5, 2000

PC Magazine December 5 2000



Search Engine Survey

PC Magazine December 5, 2000

◆ **IN THIS ROUNDUP:** 20 Search Engines.

Rating		Product	Review	Web site
●●●○○		About	Read Review	Web site
●●●○○		AltaVista	Read Review	Web site
●●○○○		AOL.com	Read Review	Web site
●●○○○		Ask Jeeves	Read Review	Web site
●●●●○		Direct Hit	Read Review	Web site
●●●○○		Excite.com	Read Review	Web site
●●●○○		FAST Search	Read Review	Web site
●●●●●	☑	Google	Read Review	Web site
●●○○○		GoTo	Read Review	Web site
●●●●○		HotBot	Read Review	Web site
●●○○○		iWon	Read Review	Web site
●●○○○		LookSmart	Read Review	Web site
●●●○○		Lycos	Read Review	Web site
●●●○○		MSN	Read Review	Web site
●●●○○		NBCi	Read Review	Web site
●●●●●	☑	Northern Light	Read Review	Web site
●●●●○		Oingo	Read Review	Web site
●●●○○		Open Directory Project	Read Review	Web site
●●●○○		Raging Search	Read Review	Web site
●●●○○		Yahoo!	Read Review	Web site

☑ **= Editors' Choice.** Ratings are on a 1-5 scale (5 is best).

INTERNET TOOLS

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NCPA
NATIONAL COTTONSEED PRODUCTS ASSOCIATION

NCPA is the trade association for the cottonseed processing industry. Products include cottonseed **vegetable oil** for cooking; **cottonseed meal**, a high protein supplement for livestock and poultry; **hulls**, a roughage for cattle feed; and **linters**, a cellulose feed stock for many industrial and consumer products.

Marketplace

COTTONSEED OIL | COTTONSEED FEED PRODUCTS

Click on the images above to buy and sell your cottonseed oil and feed products.

P. 0. Box 172267, Memphis, TN 38187-2267
Telephone 90l-682-0800
Fax 901-682-2856
E-mail: info@cottonseed.com

Click on the images below for links with more information:

Cottonseed Oil

COTTONSEED FEED PRODUCTS

Cottonseed Daily Charts and Prices
Provided by MGE

CAREER OPPORTUNITIES

Find out about an exciting career opportunity within the Cotton Industry!

We are committed to implementing new technologies that will improve the United States soybean industry in terms of market expansion and production quality. Take a look around and if you have any questions or suggestions, please let us know.

For consumer soybean information, please visit USB's Talk Soy **Project.**

The 2000 edition of Soy Stats is now available in The Library

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www.unitedsoybean.org

Agribusiness Events

AFBF Showcase

American Farm Bureau Federation Showcase

Starting Date: 01/07/01

Ending Date: 01/11/01

Deadline:

Contact:

Phone:

Place: Orlando, FL

Time: PM

Notes:

National Farm Machinery Show

Starting Date: 02/14/01

Ending Date: 02/17/01

Deadline:

Contact:

Phone:

Place: Louisville, KY

Time: AM

Notes:

Commodity Classic 2001

Annual meeting and trade show of the American Soybean Association and the National Corn Growers Association

Starting Date: 02/25/01

Ending Date: 02/27/01

Deadline:

Contact:

Phone:

Place: Henry B. Gonzalez Convention Center, San Antonio, TX

Time: AM

Notes:

Ag Publications Summit

Agricultural Publications Summit 2001

Starting Date: 07/25/01

Ending Date: 07/28/01

Deadline:

Contact:

Phone:

Place: Grand Rapids, MI

Time: AM

Notes:

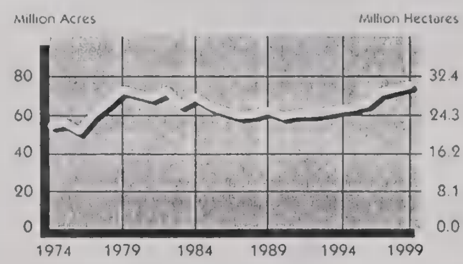
2000 Soy Stats

- Welcome
- U.S. Soy Statistics
  - Area Planted
  - Crop Yield
  - Production
  - Price to Farmers
  - Crop Value
  - Production Costs
- State Soy Statistics
- Consumption & Exports
- World Statistics

U.S. SOYBEAN STATISTICS

Previous Next

U.S. Soybean Area Planted 1974-1999



Million Acres (Million Hectares)

1974 52.5 (21.3)	1984 67.8 (27.4)	1994 61.6 (24.9)
1975 54.6 (22.1)	1985 63.1 (25.5)	1995 62.5 (25.3)
1976 50.3 (20.4)	1986 60.4 (24.5)	1996 64.2 (26.0)
1977 59.0 (23.9)	1987 58.2 (23.6)	1997 70.0 (28.3)
1978 64.7 (26.2)	1988 58.8 (23.8)	1998 72.0 (29.1)
1979 71.4 (28.9)	1989 60.8 (24.6)	1999 73.8 (29.9)
1980 69.5 (28.1)	1990 57.8 (23.4)	
1981 67.5 (27.3)	1991 59.2 (24.0)	
1982 70.9 (28.7)	1992 59.2 (24.0)	
1983 63.8 (25.8)	1993 60.1 (24.3)	

Source: USDA

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ASA American Soybean Association amsoy.org

American Soybean Association, 12125 Woodcrest Executive Dr., Suite 100, Saint Louis, Mo. USA

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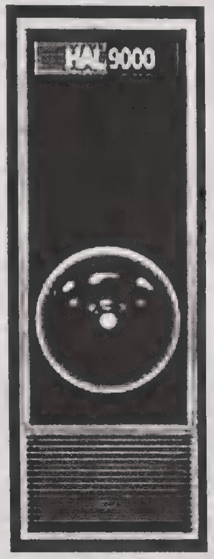

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Welcome to **amsoy.org** for 2001. For those of us who remember the classic film "*2001: A Space Oddessey*," the dawn of the third millennium provides a special opportunity to compare man's actual achievements against the science fiction concepts envisioned by Stanley Kubrick in 1968.

Today, we continue to experience rapid scientific and technological advancements in every imaginable field, including the soybean field, where modern biotechnology is providing farmers with the tools to safely produce more food, more efficiently, and with less impact on the environment.

The American Soybean Association is working to meet the challenges of the 21st century for the soy industry and for soy customers around the world.

2000-01 ASA Executive Committee

ASA is the policy advocate of the U.S. soybean farmer. Its mission is to improve U.S. soybean farmer profitability.

Hot Topics

View ASA's 2001 Policy Issues Matrix

Learn how ASA's Food Aid Initiative could save taxpayers $475 million

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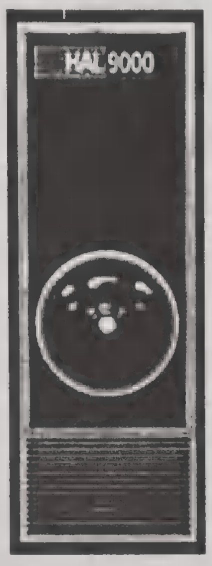

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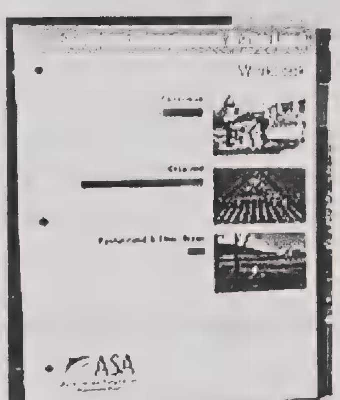
State Affiliates

Soy Health Claim Celebrates First Anniversary

Click the heart to view SoyOs Cereal, just one of many new products to hit the market since FDA approved the **Soy Health Claim**.

Environmental Program



Announcing ASA's Soybean Management & The Land Workbook and Handbook. **Learn more** about this Best Management Practices program.

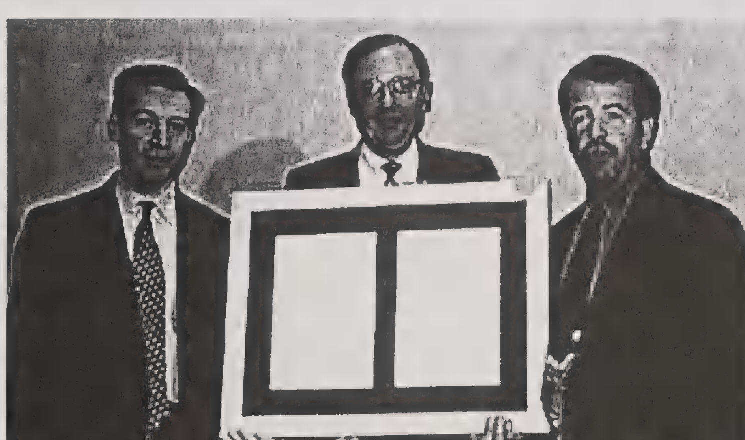
tomorrows bounty

Farmers for the Benefits of Biotechnology

PRODUCERS: Please take a few seconds to complete a survey about your biotechnology planting intentions for the 2001 season.

(AP Photo/J. Scott Applewhite)

Californian Ann Veneman, at podium, speaks after President-elect Bush announced his choices for three more cabinet posts at a ceremony in Austin, Texas, Wed., Dec. 20, 2000. Mel Martinez, left, was named head of the Department of Housing and Urban Development, Veneman was named secretary of agriculture and Don Evans, right, was named secretary of commerce. Read ASA CEO message.



ASA CEO Stephen Censky (left) and Washington Representative John Gordley (center) are presented

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Arkansas
Florida
Georgia
Illinois
Indiana
Iowa
Kansas
Kentucky
Louisiana
Michigan
Mid-Atlantic
Minnesota
Mississippi
Missouri
Nebraska
N. Carolina
N. Dakota
Ohio
Oklahoma
S. Carolina
S. Dakota
Tennessee
Texas
Virginia
Wisconsin

Coming Soon: **SoyGrower.Com**

Soy Stats Guide

A Reference Guide to Important Soybean Facts and Figures

with a signed copy of Federal legislation authorizing Permanent Normal Trade Relations (PNTR) between the United States and China. ASA President Tony Anderson (right) made the presentation at ASA's December Board of Directors meeting in recognition of the PNTR policy work by staff at ASA and state soybean affiliates. Learn more about the importance of PNTR for U.S. soybean farmers.

SPECIAL FEATURES

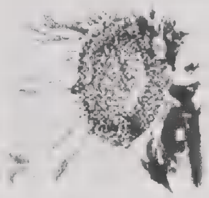
Learn more About the **History of the U.S. Soy Industry** or something about the many uses of the soybean.

Much of the information provided on this Web site is available for viewing by everyone. ASA provides no other direct services or information to the general public. To become an ASA member, use the **Online Membership Application**.

01738

National Sunflower Association

www.sunflowernsa.com



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News & Events

Especially for Producers

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The Sunflower Magazine

NuSun™

In the News...

January Grower Meetings - don't miss them!
See both the Events calendar and the related news story on upcoming grower meetings. (more)

See Agenda for upcoming NSA Research Forum
See the agenda for the upcoming NSA Research Forum to be held Jan. 17-18, 2001 at the Ramada Plaza Suites, Fargo, ND. (more)

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more

FOOD SAFETY AFIA

The AFIA Food Safety Leadership Plan

Click here to learn about food safety and biotechnology.

Industry Comments Needed on Codex Feed Definitions

December 13, 2000

The International Feed Industry Federation through the American Feed Industry Association is gathering industry's comments on proposed Codex Alimentarius feed definitions. These definitions are being used to write the Codex Code of Practice on Good Animal Feeding. A code that will present as the international feed reference point for consumers, food producers and processors, national food control agencies and the international food trade.

more

feedsearch.com version 2.0

AFIA Media Center

Below are the latest AFIA news releases.

AFIA, NGFA Announce Areas of Cooperation

- Dec 1, 2000

Industry News

New Directors Assume AFIA Posts

- Oct 24, 2000

Sixteen new directors were recently elected to the board of the American Feed Industry Association (AFIA),

E-COMMERCE EMPHASIS OF EQUIPMENT MANUFACTURERS CONFERENCE
- Oct 23, 2000

Feed Industry Exhibition Attracts Visitors Worldwide
- Oct 20, 2000

Members: Click here to download the latest *FeedGram* newsletter using Adobe Reader.

Nonmembers: Click here to download a sample *FeedGram*, the top source for the latest feed industry news.

To download Adobe Reader for free, click here.

Upcoming Events

▪ 2001 •

AFIA Board of Directors Meeting, February 5-6, Orlando, FL

Safety and Health Committee, February 12-13, Arlington, VA

Purchasing/Ingredient Suppliers Conf., February 28-March 2, San Antonio, TX

Liquid Feed Committee, March 21-22, Orlando, FL

EMC Executive Committee, March 22-23, Orlando FL

AFIA*AGRO, Mar 8-10, Jalisco, Mexico

headquartered in Arlington, Va. Elected via mail balloting by the entire association membership, the directors will serve three-year terms, commencing this month through May 2003.

Grassley, Daschle Author Separate Bills on Agribusiness Mergers, Acquisitions
- May 8, 2000

With the business community lining up in opposition, both Senate Republicans and Democrats are taking shots at agribusiness consolidation, with two separate bills aimed at curbing ag consolidations, mergers and acquisitions.

Join AFIA

AFIA is the voice of the feed industry and your company should be included. Go to the links below to see why you should become an AFIA member.

AFIA membership has many benefits. Click here to read about them.

Click here to apply for membership online.

Click here to read what AFIA members say about AFIA membership.

Click here to read about recent AFIA legislative and regulatory victories.

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Site designed and managed by the American Feed Industry Association.
1501 Wilson Blvd., Suite 1100, Arlington, VA 22209
For more information about AFIA, e-mail afia@afia.org, call (703) 524-0810 or fax (703) 524-1921.

The feed industry's single strong voice on the federal and state levels in government affairs, compliance, education and public relations.

AMERICAN FEED INDUSTRY ASSOCIATION

AFIA Action Letter Archive

This is a collection of AFIA comment action letters in PDF format. You can view the fully formatted version of each letter and print it out. You need Adobe Reader to download the letters. To download Adobe Reader for free, click here.

Download **cafo.comments.11.24.99.pdf**

To EPA. Re: Comments on the Draft Guidance Manual and Example NPDES Permit for Concentrated Animal Feeding Operations (CAFO), Nov. 24, 1999.
Last Modified on Nov 24, 1999 GMT

Download **ergo.comments.2.4.00.pdf**

Comments to OSHA on Ergo Program Standard, Feb 4, 2000.

Download **grain.8.26.98.pdf**

To the U.S. Dept. of Labor. Re: AFIA offers the following comments as part of the OSHA review of the Grain Handling Standard, August 8, 1998.
Last Modified on Nov 24, 1999 GMT

Download **guide.6.5.97.pdf**

This is the Guide for Feed Manufacturers to Comply With the FDA Final Rule Prohibiting Mammalian Protein in Ruminant
Last Modified on Nov 24, 1999 GMT

Download **lead.comments.10.29.99.pdf**

To the Office of Pollution Prevention and Toxics, EPA. Re: AFIA comments to the Notice of Proposed Rulemaking Vol. 64, No. 148 - Lead and Lead Compounds; Lowering of Reporting Thresholds; Community Right to Know Toxic Chemical Release Reporting, October 2
Last Modified on Nov 24, 1999 GMT

Download **noel.comments.6.10.99.pdf**

To Dr. Rod Noel, Chair, AAFCO Pet Food Committee. Re: Noel's memo requesting AFIA's review of proposed changes to the AAFCO Nutrient Profiles and Feeding Protocols, June 10, 1999.
Last Modified on Nov 24, 1999 GMT

Download	**Proposed 4 TAC $ 61.1 Definitions** AFIA offers these comments to the proposed rule. Last Modified on Sep 5, 2000 GMT
Download	**spcc.comments.5.7.99.pdf** To: EPA. Re: AFIA comments to "Oil Pollution Prevention and Response: Non-Transportation-Related Facilities," May 7, 1999. Last Modified on Nov 24, 1999 GMT
Download	**wellstone.comments.11.5.99.pdf** A letter sent to Sen. Wellstone in opposition to S. 1739, "Agribusiness Merger Moratorium & Antitrust Review Act of 1999." Last Modified on Nov 24, 1999 GMT

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A GLOBAL FORUM FOR THE SCIENCE AND TECHNOLOGY OF FATS, OILS, SURFACTANTS, AND RELATED MATERIALS

Quick Links

- Online Membership Directory
- AOCS Divisions
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- Online Buyers Guide

AOCS

- Welcome Joycelyn Dumas, New Accounts Receivable Clerk

AOCS Press

- New Lipids and JAOCS online!
- New Release: *Formulating Detergents and Personal Care Products* by Louis Ho Tan Tai

Meetings & Exhibits

- Annual Meeting 2001

Member Services

- Award Submission Details
- IX Congreso Seccion Latinoamericana 27-29 November 2001
- Additions and Revisions
- LPP 3rd Quarter
- "Certified" Award Winners

Click to **Search** the AOCS Web Site.

Welcome to the new, improved AOCS website. We hope you like our new look, plus several new and useful electronic initiatives.

In a recent member survey, 83 percent of AOCS members reported they receive electronic delivery of information at their office desks. This new design is in response to what we're hearing from AOCS members about how we can best serve their web-based information needs. What you'll find is a complete redesign of our site, with improved graphics and an emphasis on quick and easy navigation.

As part of our redesign, we've also launched two new web initiatives, both devised to place vital AOCS information at your mousetip: two online journals--*Lipids* and *JAOCS*--and the AOCS online membership directory. Try them and see what you think.

NOPA
NATIONAL OILSEED PROCESSORS ASSOCIATION

National Oilseed Processors Association

72nd Annual Meeting

January 26-28, 2001
Four Seasons Palm Beach Resort, Palm Beach, Florida

Click for Info and Online Registration

NOPA FACTS 2000-01
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COMPANY SERVICES SITE TOUR SOYA & OILSEED BLUEBOOK ONLINE

Soya & Oilseed Bluebook is a comprehensive directory, reference and statistical resource for the soybean and oilseed industry. Soya & Oilseed Bluebook Online is the newly expanded and fully electronic version of this valuable industry resource.

Subscribers can access information on companies that provide soybean and oilseed products, as well as equipment, supplies and services to the industry.

— **Source** both commodity and specialty ingredients including: vegetable oils, protein meals, soy protein ingredients, specialty seeds and both GMO and non-GMO products.
— **Find** equipment and services for processing and marketing.
— **Research** production, trade and utilization statistics.

Welcome to Soyatech.com

Soyatech, Inc. provides a wide range of vital services for the soybean, oilseed, and food and feed industries.

Take the site tour or see our FAQ to learn more about this world of information.

Soybean and Oilseed Industry News

Date	Headlines for January 3, 2001
1/3/2001	USDA issues oilseed and grain reports on India, Denmark, Argentina and Montenegro
1/3/2001	CBT Soy Review/ Drops on fund liquidation, soymeal
1/3/2001	Class action lawsuit filed against Aventis, manufacturers of a GM corn hybrid
1/3/2001	Mexican Reliance On U.S. Farm Exports Grows
1/3/2001	China's organic foods face international and domestic markets
1/2/2001	ISU researchers working on soybean oil proje[illegible] for Earth and beyond
1/2/2001	Canadian organic conference maps future plans
1/2/2001	Iowa Food Park adds value to soybean crop
1/2/2001	Nebraska farmers says organic farming profitable
1/2/2001	Soybeans and corn get a boost on export figures, wheat soars

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Legislative Information on the Internet

In the Spirit of Thomas Jefferson, a service of The Library of Congress

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Status of FY2001 Appropriations Bills

Consolidated Appropriations Act for FY2001 Conference Report

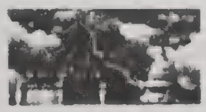
Consolidated Appropriations Act for FY2000 Conference Report

National Bipartisan Commission on the Future of Medicare.

H.R. 4328 The Omnibus Appropriations Bill for FY 1999.

FirstGov. gov: Comprehensive links to U.S.Government websites.

Historical Documents



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January 02, 2001

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Features

Ergonomics Under Fire *New!*

By *John Dyslin*

It certainly has been a tumultuous two months in Washington, D.C. A razor-thin election featuring lawsuits and appeals overshadowedOSHA's release of the ergonomics standard. With the nation held captive by an unresolved presidential election, business groups and other organizations pounced on the standard upon its release, vowing to bottle it up in court. [more...]

Health Care Ergonomic Solutions: Still a work in Progress *New!*

By *Gina Rollins*

Lost workdays due to injuries in the health care sector have declined steadily since 1992. Despite such positive statistics, however, the industry is not patting itself on the back and is implementing new ergonomic measures to reduce the numbers even further. [more...]

Highlights

The National Safety Cour
welcomes a new expert
Crossroads.

Larry Hays
Vice President, Training o
Environmental Support
Solutions. Larry's specialty
indoor air quality.

What is *Crossroads*?

Hazardous Chemical Backgrounders

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2nd Edition of the *Chemicals, Press and the Public - A Journalist's Guide to Reporting on Chemicals in the Community.*

OSHA Workers' Rights Advisor - Public Test Version. Software advisor to OSHA Act, Section 11c,

reduce the numbers even further. [more...]

EPA Eyes EPCRA Revisions *New!*
Facilities that report information under the Emergency Planning and Community Right-To-Know Act should expect to see some changes for emergency planning, release notifications, and hazardous chemical inventory reports by next Fall.

So Where Did This Stuff Come From Anyway? *New!*
We've all been there. You're sitting in some hazmat training, the instructor is passing on some bit of wisdom and you find yourself wondering, "Just where did this guy come up with this stuff anyway?".

Injury Statistics Q&A Part 4 *New!*
More than 98,000 U.S. injuries using trampolines occurred in 1999. Alan Hoskins examines product related injuries from trampolines, baby gates and lawnmowers.

From Plan to Proposal: How to Select an IAQ Consultant
For a moment, place yourself in a position many IAQ consultants find themselves. While conducting an investigation of a building you are unable to locate the cause of some classic "Sick Building Syndrome" symptoms. However, you do find a mold that has been identified as a potent developmental toxicant.

CAMEO Problems Installation Issue in Version 1.2.1
Quite a few users have complained of installation problems with the CAMEO Windows 1.2.1 updater. This is the version that repairs the defects in CAMEO Windows 1.2.

The Importance of Process Hazard Analysis

This is part one of a two part series on process hazard analysis (PHA). Part one will help define Process hazard analysis, its importance and selection needs for the team. Part two will discuss the various methodologies to perform a PHA.

Consequence data to the public.

Review Your RMP For Accuracy
Companies should take steps to double check and periodically review their Risk Management Plans to ensure that all the information is accurate and up-to-date. By year's end, RMP auditing is expected to be in full swing.

Beyond the Politics: Using the RMP to Build Community Working Relationships
Section 112 (r) of the 1990 Clean Air Act covers the prevention of accidental releases of hazardous substances into the ambient air from a stationary source.

Exposure Guidelines
Emergency response guidelines are often needed during a spill incident, in order to help determine the level of concern (LOC) in case of a possible population exposure.

Geographic Information Systems: Geography is the Common Denominator
Webster's dictionary defines geography as a "science that deals with the earth and its life; esp: the description of land, sea, air and the distribution of all plant and animal life including man and his industries."

CPR - More than just pumpin' and blowin': New CPR techniques
A family member, friend, or coworker suddenly collapses in your presence. Would you know what to do… and if so, would you do it?

Ergonomics and Behavior-Based Safety:
How some automotive companies are making it work.

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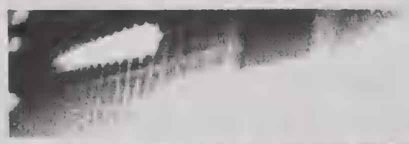

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Interest Area - Chemistry	0 listings
Interest Area - Energy	0 listings
Interest Area - Finance	0 listings
Interest Area - Mechanical	0 listings

Your areas of interest

Agriculture

Agricultural pulling machine with automatic pitch vibration damping mechanism

Methods for control and mitigation of molluscs

Methods for control and mitigation of molluscs

Chemistry

Light beam scanning read-out apparatus and recording apparatus

Edible Oil Deodorization

Adhesive transfer method

Energy

Electrochemical Deoxidation System/Technology

Security Monitoring System Technology

Electrostatically enhanced separator (EES)

Finance

System for Managing Intellectual Property Assets and Agreements

Web-based Method for Auctioning Products based on Actual Costs.

System for automating the sourcing of products.

Mechanical

Maneuvering prediction program at initial design stage of a ship

A search can lead to an introduction. Learn more here.

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Search Tips:

To find all the conditions in the search field, use <AND> between words or terms.

To exclude a condition in the search field, use <NOT> between words or terms.

Put * after a word to find all endings. *Oil** to find *oil, oils, oiler, oiling,* etc.

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displaying **(1 - 12)** of **15** listings found.
1 2 [Next]

84% **Container for foods**
An edible food container is provided with two different fatty coating layers including a top glaze that is free of sugar.

83% **Low fat baked snacks**
A process has been developed which can be used to provide regular, well registered, non overlapping slices from irregular shaped raw ingredients.

82% **Coatings for Non-woven Materials and Textiles for Skin Health and Comfort**
This innovative technology describes a petrolatum lotion formulation that can be used as a coating for absorbent articles with topsheets such as diapers, as well as bandages, nylon hosiery and clothing to reduce skin irritation and rashes, in addition to increased wearer comfort, via continuous ointment delivery onto the skin surface via normal contact, wearer motion and/or body heat.

82% **Low Cost, Renewable Resource Surfactant**
This innovative technology provides low cost Methyl Ester Sulfonate (MES) surfactants, which are heavy-duty renewable resource surfactants (not based on petroleum), with excellent color and purity, without the use of older technology involving a bleaching step.

80% **Edible Oil Deodorization**
The invention relates to a process for

deodorizing edible oils or fats using inert gas inplace of steam stripping.

80% **Deodorizing edible oil and/or with non-condensible inert gas and recovering a high quality fatty acid distillate**
The invention relates to a process for deodorizing edible oils and/or fats comprising: heating edible oil and/or fat to an elevated temperature; introducing or injecting non-condensible inert gas into said edible oil and/or fat to strip or remove substances that impart disagreeable odor and taste to said edible oil and/or fat; and recovering the resulting deodorized edible oil and/or fat, wherein an amount of said non-condensible inert gas introduced or injected is substantially less than the...

80% **Method of recovering waste heat from edible oil deodorizer and improving product stability**
The invention relates to a process for recovering heat from deodorized edible oils and stabilizing the same comprising: introducing or injecting non-condensible inert gas into hot deodorized edible oil as the hot deodorized edible oil is cooled by indirectly heat exchanging with crude oil to be deodorized.

80% **Oxabicycloalkane Herbicides**
New types of chemical compounds were developed which can prevent the growth of grassy weeds and broad-leafed plants in agricultural fields.

77% **Synthetic (lubricating) fluids with built-in antioxidant function**
The fluids described allow lubricants to be formulated in a novel and effective way that can provide excellent resistance to degradation in high temperature operating environments.

77% **Natural Grip, High Force, Liquid Product Dispenser**
This invention is a new liquid sprayer having a

more comfortable grip and delivering higher forces to the pump, while maintaining lower actuation forces for the consumer than traditional sprayers which, in turn, could increase sales and profits from higher consumer satisfaction and the ability to dispense high viscosity liquids that are very difficult to spray using typical devices.

77% **Extraction of Valuable Materials from Plant Matter**
A package of technologies useful in extracting valuable materials from plant matter.

77% **Cooking on glass-ceramic plates with halogen lamps**
Cooking at normal atmospheric pressure makes possible the use of light constructions of the vessels, in particular glass vessels, which in turn permit the use of halogen lamps as heating elements in a glass-ceramic plate.

---

1 2 [Next]

Cambridge, MA, USA Manchester, United Kingdom Tokyo, Japan Seoul, Korea

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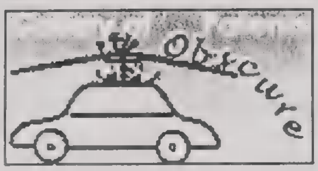

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Headlines

- Delphion announces new business model and features
- PatentLab-II: Powerful tools...instant analysis
- ISI article links for non-patent citations
- INPADOC data now on IPN

More site news ...

PACIFIC NORTHWEST EDITION

January 04, 2001

Today's Headlines

Today's Ag Radio

From The Editor
We hope you enjoy our site and today's Ag programs. Please contact us with any questions. More

Line on Agriculture
January 04, 2001 - Secretary Glickman looks back and to the future. More

Northwest Report
January 04, 2001 - Congress is back in session and talking tax cuts. Cattlemen are suing Secretary of Agriculture Dan Glickman. More

Market Line
January 04, 2001 - Wheat sharply higher on fund buying More

Fruit Grower Report
January 04, 2001 - Harvesting Red Delicious apples for taste or storage. More

Northwest Livestock Report
January 04, 2001 - LMA sues Ag Secretary over inaction on beef checkoff referendum More

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January 04, 2001 - LMA sues Ag Secretary over inaction on beef checkoff referendum More

Northwest Farm and Ranch Report
January 04, 2001 - USDA economist expects lower wheat acreage More

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GEMPLER'S This premier on-line catalog offers over 10,000 hard-to-find, industrial-grade products for those working primarily in agriculture, horticulture, construction and grounds maintenance.

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January 2001

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Headlines

3:34 p.m. EST, Thursday, January 4, 2001

What's News—

* * *

Business and Finance

BIG RETAILERS registered disappointing performances in December. Wal-Mart's sales were flat, while Sears posted falling sales and said it would close 87 stores.

* * *

Lehman Brothers reported a 33% increase in earnings, while Bear Stearns Co.'s earnings dropped 6%. However, profits at both financial services companies easily topped analysts' estimates as their revenues strengthened.

* * *

Stocks pulled back Thursday following the previous session's strong rally as investors remained cautious about the economic outlook in the wake of the Fed's surprise interest-rate cut.

* * *

Lockheed Martin's F-22 fighter is facing more political hurdles to its development after a Defense Department official recommended putting off production of the aircraft for at least nine months and possibly a year.

* * *

Sotheby's former chairman is expected to announce he has

Markets 3:47 p.m. EST

DJIA	10899.09	-46.66
S&P 500	1331.69	-15.87
Nasdaq	2565.18	-51.51
Nikkei 225*	13691.49	-94.20
DJ Stoxx 50*	4534.100098	+77.12999

*at market close Source: Reuters

WORLD-WIDE

Arab Leaders Issue Demands to Arafat On Mideast Talks

Arab foreign ministers insisted that Palestinian refugees' right of return is "sacred," leaving Arafat little room to maneuver on a key concession demanded in a U.S. peace plan.

PAGE ONE

Greenspan's Action Reflects Rising Fear Of a U.S. Recession

The Fed cut a key interest rate, sending markets soaring. The central bank, in a rare move between meetings, lowered the federal funds target to 6% from 6.5%.

California's PG&E

Year-End Review Markets & Finance

See a year-end review of world financial and news events. Our enhanced coverage includes an interactive timeline, a roundtable of analysts discussing the economy's outlook and new online discussion of the year ahead.

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hired Credit Suisse First Boston to explore strategic options for his controlling stake in the auction house.

* * *

Service-sector growth slowed considerably last month in the U.S., with the purchasing managers' index dropping to 53 from 58.5. Separately, factory orders climbed 1.7% in November, largely due to higher demand for aircraft.

* * *

True North Communications said it will record a fourth-quarter charge related to the advertising agency's loss of the DaimlerChrysler account.

* * *

Toys "R" Us reported that same-store sales rose 3.5% for the nine-week holiday-shopping period. Shares of the toy retailer surged on the news.

* * *

Delta is trimming its flight schedule an average of 2.7% through the first quarter as it struggles to manage continuing pilot shortages stemming from a contract dispute.

* * *

Mercata plans to shut down, citing an inability to raise new funds. The online group-buying service pulled its IPO plans earlier in the week.

* * *

Engage plans to cut its work force about in half as part of a broad restructuring at the online-

Gropes to Survive Electricity Squeeze

A look inside California electricity giant PG&E shows a business struggling to navigate a once-familiar world turned topsy-turvy.

Gates Is a Style Icon In Korean Fashion, Where Geek Is Chic

Bill Gates may not be considered the epitome of chic in America, but in South Korea the Microsoft founder is a serious style icon.

CAPITAL

Voters are starting to demand more spending on education, and while parents have always wanted better schools, the big change now is that they're willing to dig deeper into their own pockets.

ALSO IN TODAY'S EDITION

Marketplace

These two laptops are no lightweights.

Tech Center

Showcased in

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Dow Jones & Co.

Corrections

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marketing company, a unit of CMGI.

* * *

Many economists expect corporate profits to decline for the first time since 1998 despite the Fed rate cut, and some forecast a profits recession.

* * *

The Seattle Times reached a tentative settlement with workers in their 45-day strike against the newspaper.

* * *

Ford is seeking to settle all the individual U.S. suits resulting from rollovers of Explorers with Firestone tires. The firm settled four more cases and is close to settling a fifth.

* * *

California regulators proposed a temporary surcharge on retail power bills, but the increase fell far short of what utilities say is needed to keep solvent. Consumer groups also criticized the move.

* * *

U.S. auto sales fell 8% in December, the second straight month of decline, ending a record sales year. Car makers blamed the economic slowdown and said they were cautious about 2001.

* * *

Goldman Sachs and Morgan Stanley ranked as the top two merger advisers in 2000. Merrill fell to fourth from second place in 1999; CSFB ranked third.

* * *

2000: Where are they now?

Politics & Policy

Ashcroft nomination may be test for Bush.

Money & Investing

Time for offense, or more defense?

Editorial Page

Greenspan can't do it on his own.

Leisure & Arts

Eric Gibson on Sol LeWitt

Sports

See the latest sports news from Total Sports.

Voices

The Market's Direction
Tech's Future
Potter Sites
Crackdown

The ECB left its key interest rate unchanged as expected at 4.75%, reflecting the euro zone's comparatively bouyant economic outlook.

* * *

Federal-Mogul plans to fight asbestos litigation rather than file for Chapter 11 bankruptcy protection, citing $550 million in new credit lines.

* * *

Network Associates named technology veteran George Samenuk as chief executive officer of the maker of security and anti-virus software, which recently surprised investors with a loss and executive departures

* * *

Intel introduced a faster version of its low-cost Celeron chip for personal computers and also introduced a cheaper, though slightly slower, Pentium 4 chip for more expensive desktop computers.

* * *

Letsbuyit.com's management board resigned as the European e-commerce company faces insolvency proceedings.

* * *

Microsoft was charged with racial discrimination in a suit in which the plaintiffs' lawyers may focus on the issue of merit pay.

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Volume 2, Issue 1.1
January 2001

EPA United States Environmental Protection Agency

"...to protect human health and to safeguard the natural environment..."

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For KI S

FIRSTGOV

"The new standards will prevent 8,300 premature deaths, 5,500 cases of chronic bronchitis in adults and 17,600 cases of acute bronchitis in children."
---Administrator Carol M. Browner announcing new diesel standards, December 21, 2000

Headlines

New Standards Set For Levels In Paint, Dust, and Soil
EPA has set new standards to identify dangerous levels of lead in paint, dust and soil that will, for the first time, provide home owners, school playground administrators, childcare providers and others with standards to protect children.

First-Ever Water Quality Criteria For Nutrients
For the first time, EPA is setting criteria to protect waters from excessive nutrients that can lead to Pfisteria and red tide and result in fish kills and potentially harmful human health effects.

Pollution From Heavy-Duty Trucks and Buses to be Cut by 95%

The cleanest running heavy-duty trucks and buses in history--95 percent cleaner than those today--will result from new EPA action. In addition, sulfur in diesel fuel will be reduced by 97 percent.

Other Stories

EPA to Stop Air Pollution From Coal-Fired Power Plants
In an ongoing initiative to address illegal air emissions from coal-fired power plants, EPA has announced a record settlement with Cinergy, an electric utility based in Cincinnati. The $1.4 billion enforcement action is the largest ever under the Clean Air Act.

EPA Plans to Regulate Power Plant Mercury Emissions
To reduce health risks, EPA plans to regulate emissions of mercury and other toxics from power

Announcements

This Month in EPA History
Activities Update
What's New on the EPA Web

Features

Speeches & Testimony
Carol M. Browner, Administrator
-- Remarks Prepared for Delivery Diesel Announcement Washington, DC December 21, 2000

Education Matters
Office of Environmental Education

EPA USA
Texas and Minnesota
Hudson River
California

Emerging Issues
Brownfields Communities get $1.8M for Job Training

National Environmental Achievement Track

Brownfields Job Training Expands

plants fired by coal and oil. Proposed rules will be issued in 2003.

EPA Headquarters Press Releases

Local Stories

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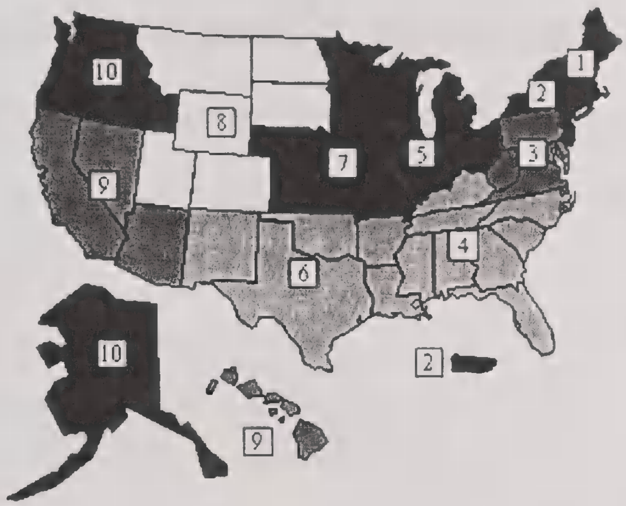

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Region 9 - Arizona, California, Hawaii, Nevada, and Pacific Islands and Tribal Nations subject to US law.
Region 10 - Alaska, Idaho, Oregon, and Washington.
ORD - Office of Research and Development.

People & Profiles
Veteran Protectors: EPA's 1970 Pioneers

The Early Days at EPA

Consumer News
Asbestos in Attic Insulation

Pesticide Diazinon Use Being Phased Out

Green Products: Environmentally Preferable Purchasing

Public Participation
National Advisory Council for Environmental Policy and Technology

Public Involvement in Environmental Permits: A Reference Guide

Money Matters & Jobs
Employment Opportunities at EPA and in the field of Environmental Protection:

Recent Publications
Terms of The Environment

Environmental History IQ
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EPA United States Environmental Protection Agency

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PART I. FACILITY IDENTIFICATION INFORMATION *(FORM R)*

DOCUMENT CONTROL NUMBER: 1398120182175

Section 1. Reporting Year

Reporting Year: 1998

Section 2. Trade Secret Information

2.1 Trade Secret: NO

2.2 Sanitized Copy: Unsanitized

Section 3. Certification

CERTIFYING OFFICIAL'S NAME	CERTIFYING OFFICIAL'S TITLE	CERTIFYING OFFICIAL'S SIGNATURE	DATE SIGNED
JOHN MULHOLLAND	PLANT SUPERINTENDENT	No Data	25-JUN-99

Section 4. Facility Identification

TRI Facility ID: 23324CRGLL501BA

4.1 Facility Name and Address.

Facility Information

NAME	STREET	CITY	COUNTY	STATE	ZIP CODE
CARGILL INC.	501 BARNES RD.	CHESAPEAKE	CHESAPEAKE CITY	VA	23324

KK-56

Mailing Information

NAME	STREET	CITY	COUNTY	STATE	ZIP CODE
CARGILL INC.	P.O. BOX 7506	CHESAPEAKE		VA	23324

4.2 Facility Classification

ENTIRE FACILITY	PARTIAL FACILITY	FEDERAL FACILITY
YES	NO	NO

4.3 Technical Contact

Not Available to the Public as this information is only for Intranet.

4.4 Public Contact

NAME	PHONE
JOHN MULHOLLAND	7574945582

4.5 SIC Codes

SIC CODE	SIC CODE DESCRIPTION
2075	SOYBEAN OIL MILLS

4.6 Location

LATITUDE	LONGITUDE
364-47-30	076-17-04

4.7 Dun & Bradstreet Numbers

DUNS NUMBER
063425359
NA

4.8 RCRA ID Numbers

RCRA ID NUMBER
NA
VAP000009081

4.9 NPDES Permit Numbers

NPDES PERMIT NUMBER
NA
VA0004448

4.10 Underground Injection Well Code (UIC) ID Number

UIC ID NUMBER
NA

5 Parent Company Information

Parent Company Name: NA

Parent Company DUNS Number: NA

PART II. CHEMICAL - SPECIFIC INFORMATION

DOCUMENT CONTROL NUMBER: 1398120182175

Section 1. Toxic Chemical Identity

1.1 CAS Number: 000110543

1.2 Toxic Chemical or Chemical Category Name: N-HEXANE

1.3 Generic Chemical Name: NA

Section 2. Mixture Component Identity

2.1 Supplier Provided Generic Chemical Name: NA

Section 3. Activities and Uses of the Toxic Chemical

3.1 Manufacture the Toxic Chemical:
Produce: NO Import: NO On-Site Use/Processing: NO
Sale/Distribution: NO Byproduct: NO Impurity: NO

3.2 Process the Toxic Chemical:
Reactant: NO Formulation Component: NO Article Component: NO Repackaging: NO

3.3 Otherwise Use the Toxic Chemical:
Chemical Processing Aid: YES Manufacturing Aid: NO Ancillary or Other Use: NO

Section 4. Maximum Amount of the Toxic Chemical Onsite During the Calendar Year

Maximum Chemical Amount: 100,000 - 999,999 pounds

Section 5. Quantity of the Toxic Chemical Entering each Environmental Medium Onsite

5.1 Fugitive or Non-Point Air Emissions

NA	TOTAL RELEASE (pounds/year)	BASIS OF ESTIMATE
NO	22000	C - MASS BALANCE

5.2 Stack or Point Air Emissions

NA	TOTAL RELEASE (pounds/year)	BASIS OF ESTIMATE
NO	293000	C - MASS BALANCE

5.3 Discharges to Receiving Streams or Water Bodies

STREAM/WATER BODY NAME	TOTAL RELEASE (pounds/year)	BASIS OF ESTIMATE	% FROM STORMWATER
NA			0

5.4.1 Underground Injection Onsite to Class I Wells.

NA	TOTAL RELEASE (pounds/year)	BASIS OF ESTIMATE
NO DATA	NO DATA	NO DATA

5.4.2 Underground Injection Onsite to Class II-V Wells.

NA	TOTAL RELEASE (pounds/year)	BASIS OF ESTIMATE
NO DATA	NO DATA	NO DATA

5.5 Disposal to Land Onsite

5.5.1A RCRA Subtitle C Landfills

NA	TOTAL RELEASE (pounds/year)	BASIS OF ESTIMATE

YES		

5.5.1B Other Landfills

NA	TOTAL RELEASE (pounds/year)	BASIS OF ESTIMATE
YES		

5.5.2 Land Treatment/Application Farming

NA	TOTAL RELEASE (pounds/year)	BASIS OF ESTIMATE
YES		

5.5.3 Surface Impoundment

NA	TOTAL RELEASE (pounds/year)	BASIS OF ESTIMATE
YES		

5.5.4 Other Disposal

NA	TOTAL RELEASE (pounds/year)	BASIS OF ESTIMATE
YES		

Section 6. Transfers of the Toxic Chemical in Wastes to Off-Site Locations

6.1 Discharges to Publicly Owned Treatment Works (POTWs)

6.1.A Total Quantity Transfered to POTWs and Basis of Estimate

6.1.A.	TOTAL TRANSFERS (pounds/year)	BASIS OF ESTIMATE
1	500 - 999	C - MASS BALANCE

6.1.B POTW Locations

6.1.B.	POTW NAME	ADDRESS	CITY	STATE	COUNTY	ZIP CODE
1	HAMPTON ROADS SANITATION DISTR VIRGINIA INITIATIVE PLANT	FOOT WEST 44 ST.	NORFOLK	VA	UNKNOWN	23508

2	NA						

6.2 Transfers to other Off-Site Locations

RCRA Number: DEQ#417
Name: SUFFOLK REGIONAL LANDFILL
City: SUFFOLK
County: UNKNOWN
Parent Company Controlled: YES
Address: 1 BOB FOELLER DR.
State: VA
Zip Code: 23434

TOTAL TRANSFERS (pounds/year)	BASIS OF ESTIMATE	WASTE MANAGEMENT TYPE
1 - 10	M - DATA MONITORING OR MEASUREMENTS	M72 - Landfill/Disposal Surface Impoundment

Section 7A. On-Site Waste Treatment Methods and Efficiency

7A.1a. Waste Stream: WASTEWATER

7A.1b.	WASTE TREATMENT METHOD(S) SEQUENCE
1	P42 - STRIPPING -- STEAM
2	NA

7A.1c. Range of Influent Concentration: 100 PPM TO 1 PERCENT
7A.1d. Waste Treatment Efficiency Estimate: 97
7A.1e. Based on Operating Data?: NO

Section 7B. On-Site Energy Recovery Processes

ON SITE ENERGY RECOVERY PROCESSES
NA

Section 7C. On-Site Recycling Processes

ON SITE RECYCLING PROCESSES
NA

Section 8. Source Reduction and Recycling Activities

SECTION	TYPE OF QUANTITY	PRIOR YEAR	CURRENT REPORTING	FOLLOWING YEAR	SECOND FOLLOWING

			YEAR		YEAR
8.1	Quantity Released	360000	315000	360000	3600[illegible]
8.2	Quantity Used for Energy Recovery Onsite	NA	NA	NA	NA
8.3	Quantity Used for Energy Recovery Offsite	NA	NA	NA	NA
8.4	Quantity Recycled Onsite	NA	NA	NA	NA
8.5	Quantity Recycled Offsite	NA	NA	NA	NA
8.6	Quantity Treated Onsite	NA	NA	NA	NA
8.7	Quantity Treated Offsite	600	600	600	600

8.8 One-Time Event Release: 0 pounds

8.9 Production Ratio: 81.92

8.10 Source Reduction Activities

SOURCE REDUCTION ACTIVITIES	METHOD 1	METHOD 2	METHOD 3
NA			

8.11 Additional Data Indicator: NO

TRI *Explorer* v3.0

EPA Office of Environmental Information

Release: Industry Report

See Note Return to selection

TRI On-site and Off-site Reported Releases (in pounds), N-HEXANE By Industry, State of Ohio, 1998

Row #	Industry	Total Air Emissions	Surface Water Discharges	Underground Injections	Releases to Land	Total On-site Releases	Total Off-site Releases	Total On- and Off-site Releases
1	20 Food	1,940,270	0	0	0	1,940,270	0	1,940,270
2	26 Paper	11,272	0	0	0	11,272	53	11,325
3	27 Printing	2,080	0	0	0	2,080	0	2,080
4	28 Chemicals	155,032	0	0	0	155,032	3	155,035
5	29 Petroleum	51,116	0	0	5	51,121	250	51,371
6	30 Plastics	312,090	0	0	0	312,090	250	312,340
7	34 Fabricated Metals	186,003	0	0	0	186,003	0	186,003
8	35 Machinery	500	0	0	0	500	0	500
9	36 Electrical Equip.	5	0	0	0	5	0	5
10	37 Transportation Equip.	25,589	0	0	0	25,589	0	25,589
11	39 Miscellaneous	.	.	.	.	.	.	.
12	Multiple Codes 20-39	1,740	0	0	0	1,740	0	1,740
13	Combination New/Original Ind.	1,261	0	0	0	1,261	0	1,261
	Original industry subtotal:	2,686,958	0	0	5	2,686,963	556	2,687,519
14	5169 Chemical Wholesalers	8,341	0	0	0	8,341	0	8,341
15	5171 Petroleum Bulk Terminals	20,409	7	0	306	20,722	2,724	23,446
16	4953/7389 RCRA/Solvent Recovery	4,517	2	0	0	4,519	584	5,103
	New industry subtotal:	33,267	9	0	306	33,582	3,308	36,890
	Total	**2,720,225**	**9**	**0**	**311**	**2,720,545**	**3,864**	**2,724,409**

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Create comma-separated values, compatible with spreadsheet and databases.

Download all records

View other report type:

- Transfers Off-site for Further Waste Management; or
- Quantities of TRI Chemicals in Waste (waste management)

Note: Reporting year (RY) 1998 is the most recent TRI data available. Facilities reporting to TRI were required to submit RY 1998 data to EPA by July 1999. TRI Explorer is using a *"frozen"* data set that includes revisions submitted to EPA as of March 29, 2000 for the years 1988 to 1998 (i.e., revisions submitted to EPA after this time are not reflected in TRI Explorer reports). Please access EPA Envirofacts to view TRI data with the most recent revisions.

On-site releases are from Section 5 of the Form R. Off-site releases are from Section 6 (transfers off-site to disposal) of the Form R. Off-site releases include metals and metal compounds transferred off-site for solidification/stabilization and for waste water treatment, including to POTWs.

A decimal point, or "." denotes the following:

1. if a decimal point is reported across an entire row, the facility submitted a Form A (i.e., the facility certified that its total annual reportable amount is less than 500 pounds, and does not manufacture, process, or otherwise use more than 1 million pounds); or
2. if a decimal point is reported in a single column, the facility left that particular cell blank in its Form R submission (a zero in a cell denotes either that the facility reported "0" or "NA" in its Form R submission).

Users of TRI information should be aware that TRI data reflect releases and other waste management of chemicals, not exposure of the public to those chemicals. Release estimates alone are not sufficient to determine exposure or to calculate potential adverse effects on human health and the environment. TRI data, in conjunction with other information, can be used as a starting point in evaluating exposures that may result from release and other waste management activities that involve toxic chemicals.

Release: January 9, 2001
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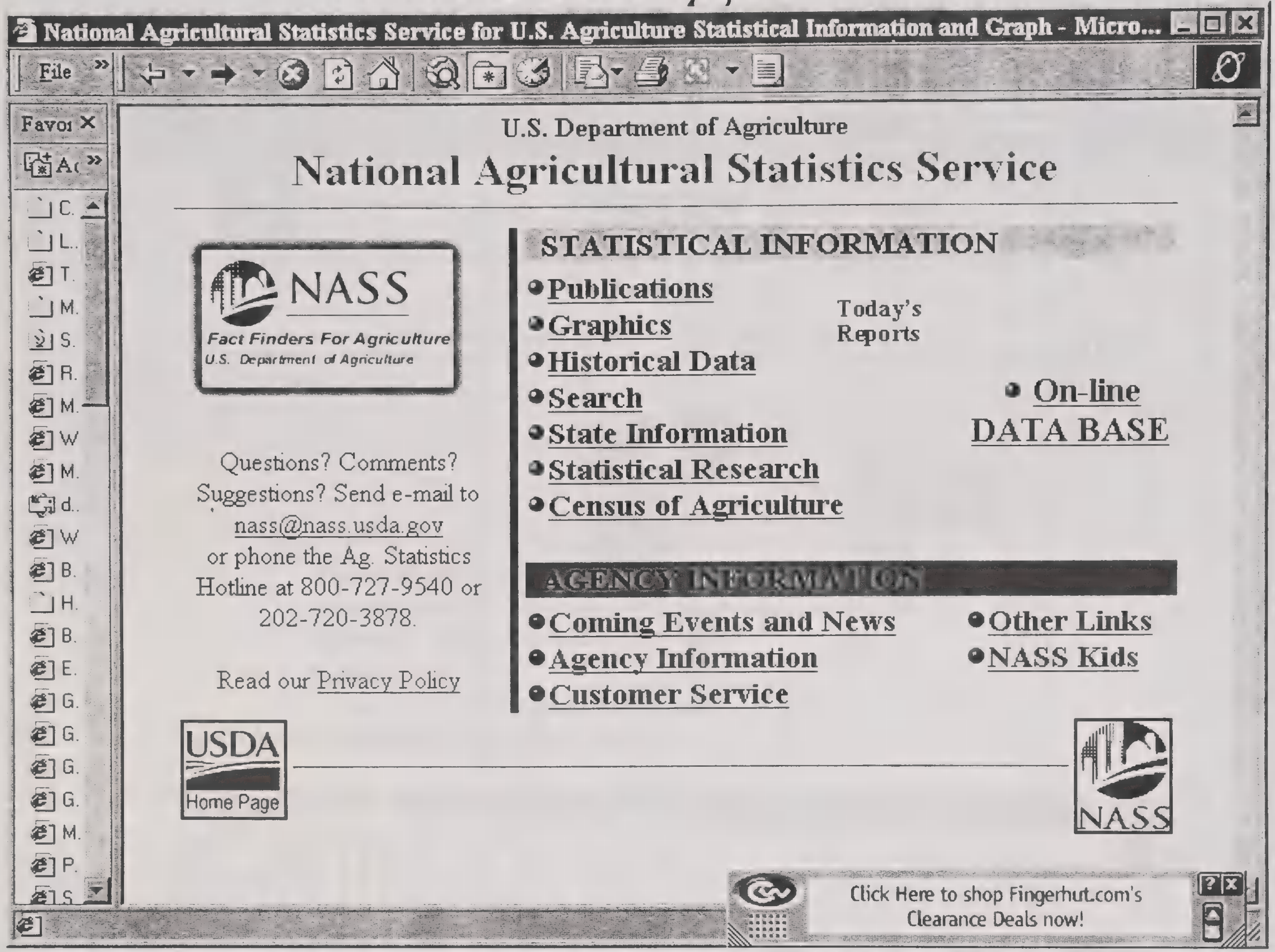

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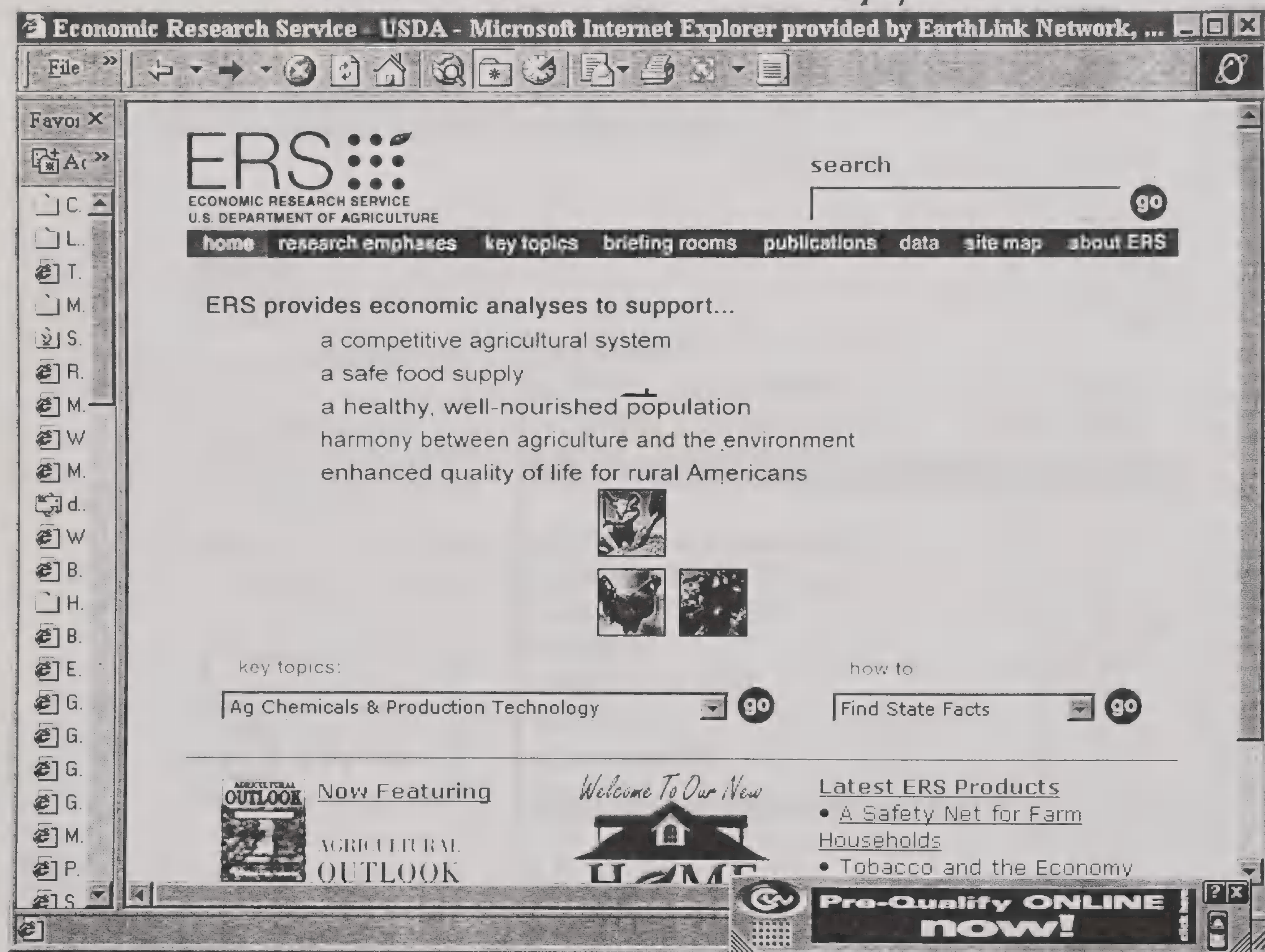

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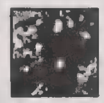

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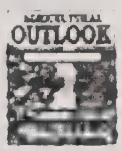

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Fishing Wheatley 21st Century Tan Fly Box NEW	**$11.50**	3	01/11 14:07
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Tools for Improving Profits

January 21–23, 2001 ✸ Hyatt Regency New Orleans

Hiring and Retaining Good Employees

Patricia Bivins
Equal Opportunity Commission
New Orleans, LA

Hiring and Retaining Good Employees

Patricia T. Bivins, District Director
U.S. Equal Employment Opportunity Commission
New Orleans District Office

Discussion Points

Why emphasize employee retention?

When should retention planning begin?

What should be considered in a well-rounded retention program?

Why Emphasis on Retention?

Needs of employees have changed.

Opportunities available.

Needs of companies have changed.

To be productive companies need

Workforce that is:

Stable.

Talented.

Ideas turn into productive final products.

What Should You Do?

"... turn employees into raving fans of the organization they [work] for."

Gung Ho! Ken Blanchard & Sheldon Bowles

How Can I Get “Raving Fans?”

Management should

Demonstrate a strong leadership COMMITMENT.

Possess knowledge of the status of the organization today.

How Can I Get "Raving Fans?"

Management should

Ensure strong COMMITMENT to resources:

- People
- Funding

Why Commitment?

Management commitment leads to

Employee commitment.

Employee commitment

Reduces the cost of transition

Advertising for vacancies.

Training new employees.

Lost of production due to unskilled employees.

How do you get commitment?

Designing and implementing an effective, well-rounded retention program.

What should be considered when designing a well-rounded retention program?

Recruiting & hiring talented workers.
Regardless of race, creed, color, or age.
Expand talent search to under utilized populations.

What should be considered when designing a well-rounded retention program?

Training - Developing the skills possessed by these talented workers, and

What should be considered when designing a well-rounded retention program?

Transforming- Make the worker a team member. Request and include ideas of workers to improve products.

Practices Used to Help Retain Employees

"Finding & Keeping Great Employees"
Jim Harris & Joan Brannick

1. Engage the soul.
2. What gets rewarded gets done.
3. More than money.

Practices continued

4. Learning Drives Earning.
5. Get a Life.
6. In the Loop.

Practices

7. Lighten Up.
8. Free at Last.

Last But Not Least

Equal Opportunity

Ensure employment practices are:

- Communicated by management.
- Applied equally for entire organization.
- All levels of the organization know the policies.

Summary

Retention Program

Begins with recruitment & hiring.

Provides opportunities for skill development.

Encourages a team effort.

Ensures equal opportunity for all.

U.S. Equal Employment Opportunity Commission

New Orleans District Office

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Partnerships to Reduce Pollution from Agricultural Activities

Carlton Dufrechou
The Lake Pontchartrain Basin Foundation
Metairie, LA

Partnerships to Reduce Pollution from Agricultural Activities

The Lake Pontchartrain Basin in a 5,000 square mile watershed in southeast Louisiana. The Basin encompasses barrier islands, coastal wetlands, the densely populated metro New Orleans area, and rural woodlands and farmlands. For the last 50 years, Pontchartrain's water quality and habitats have experienced significant degradation primarily due to activities of man. Adverse activities include: dredging of wetlands, municipal stormwater discharges, poorly operated/inadequate wastewater treatment plants, agricultural runoff and others. The principal problem in the rural northern portion of the Pontchartrain Basin is runoff from agricultural activities, primarily dairies.

The Lake Pontchartrain Basin Foundation (LPBF) was created by the Louisiana legislature in 1989 to restore and preserve the environmental health of the Pontchartrain Basin. The LPBF is a private, non-profit organization that attempts to use innovative and non-regulatory approaches where feasible to address pollution problems.

From the early 1990's the LPBF has partnered with the agricultural industry to establish environmental improvement programs. In 1993, the LPBF partnered with the Soil Conservation Services (currently Natural Resource Conservation Service (NRCS)), the Louisiana Farm Bureau (LFB) and individual farmers to create a cost share program to help fund construction of livestock waste retention lagoons. Over a five year period, 67 lagoons were constructed. In 1998, the LPBF partnered with the Bogue Chitto-Pearl River Soil and Water Conservation District to develop a conservation tillage program. This program encourages farm management practices to reduce soil erosion and conserve energy through reduced tillage practices, soil conservation practices, and reduction of chemical fertilizer-nutrient wastes. This ongoing partnership provides conservation tillage equipment which is available for use by district farmers. Most recently, the LPBF partnered again with the NRCS, LFB, and livestock operators to create a waste lagoon clean out program. This program is designed as a model to encourage ongoing maintenance of waste retention lagoons. Since program announcement in December 2000, 10 farmers have applied for the partnerships.

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Incident Database and Macroanalysis to Help Set Safety Direction

John A. McIntosh, III
Procter & Gamble
Cincinnati, OH

Incident Database and Macroanalysis to Help Set Safety Direction

Introduction

When safety incidents occur, most organizations conduct investigations and prepare reports in an effort to learn the cause and to determine how to prevent similar incidents from occurring again. What happens to the collected information? Is the data reviewed frequently? If incidents occur in similar systems, do reviews of the data reveal lessons learned? Answers to these questions may depend on how easily the data can be accessed. Databases offer an effective option for managing large amounts of information. Used to study process safety trends and underlying causes of incidents, databases can be powerful and effective risk management tools. Macroanalysis of incident data can reveal process safety weaknesses and help risk managers determine where to focus effort and resources. Three case studies illustrate how Corporate process safety personnel, in conjunction with product category personnel, use incident data to reveal process safety weaknesses; to initiate equipment design modifications and procedural changes; and to improve risk management programs.

Background

A main objective of the corporate process safety organization is to improve the understanding of risks associated with processes and to reduce the likelihood and consequences of incidents. One strategy is to study incidents and reapply lessons learned. Incident reports have been collected at P&G for almost 20 years to gather information and report findings. Originally, incident reports were used primarily for documentation, but not much was done with the data in the reports. Manually reviewing reports and sorting incidents was not an effective method for analysis of incident data. Consequently, little data analysis was done, and potential lessons learned may not have been revealed. To improve data analysis capabilities, a process safety incident database was designed and developed. The process safety incident database provides an effective tool for managing the large amounts of information found in incident reports.

Data Collection

What data is important? How will the data be analyzed? What do we want to learn from the data? Answers to these questions help define what data to collect and how to collect it. For process safety incident analysis, the first step is to define what scenarios should be considered process safety incidents. This streamlines the contents of the database, and allows the analyst to focus on scenarios of interest. Procter and Gamble defines a process safety incident as "anything in a process or utility system which caused or could have caused a fire; an explosion; a release of flammable, reactive or hazardous material; or an overpressure condition (positive or negative)".

To develop an accurate and complete picture of process safety history, all incidents need to be investigated and reported. Consistent data reporting from process to process and from site to site maximizes the usefulness of the data. To ensure consistency in data collection and reporting, Procter and Gamble uses a standard incident investigation report form. The incident investigation form contains predefined data fields, including process, equipment, materials, costs, and incident category. The predefined data fields provide consistency and allow for easy queries on specific pieces of data. The report also contains sections for more detailed description of the incident and the causes. These sections provide more detailed information which may reveal critical insight on the incident.

Data Integrity

Incident investigations and reporting are critical components of process safety management. To reduce the likelihood and consequences of incidents, lessons learned from process safety incidents should be reapplied to similar systems. To do this, we need a complete, company-wide picture of process safety incidents. How do we ensure all process safety incidents are reported? How do we ensure data on the incident reports are complete and accurate? At Procter and Gamble, all risk program leaders are trained in conducting incident investigations and reporting results, including training on the use of incident investigation report forms. Following an incident, site personnel and corporate risk managers review incidents and discuss report content to ensure corporate risk managers fully understand the incident and its causes. Any unclear information or questions are resolved before database entry. Incident reporting is also checked via risk management program audits. Incident reporting is a specific line item in the audit. Auditors compare the number of incidents reported during a specific period of time and compare this with site records.

Database Design and Future Enhancements

The original Procter and Gamble process safety incident database was developed using commercially available, PC-based database software. Each field in the incident database corresponded to a field on the incident report form. The database structure provided the analyst with flexibility to perform ad hoc queries in addition to producing predefined reports. Flexibility to perform queries on any of the data fields is crucial. This allows the analyst to probe more deeply into cause and effect relationships. The next generation incident database is being developed on commercially available, mainframe-based software. While maintaining the flexibility of the original database, the change in software will align the incident database with our existing industrial health and safety database. This alignment will allow sites to enter data directly into the incident

database, eliminating the need for a "hard copy" to be filled out and re-entered by corporate process safety.

Database Uses

The following Case Studies illustrate how Corporate process safety personnel, in conjunction with product category personnel, use the incident data to reveal process safety weaknesses, improve safety programs, and focus resources effectively.

Case Study 1: Risk Reduction -- Reactive Releases

As we have pointed out, Procter and Gamble defines an incident as "Anything in a process or utility system which caused or could have caused a fire, explosion, release of flammable, reactive or hazardous material, or an overpressure condition (positive or negative)." The first step in dealing with large numbers of incident reports is to assure, as much as possible, that all incidents are classified into one of these defined categories. Figure 1 is a Pareto chart showing the distribution of the numbers of incidents in each of the defined categories.

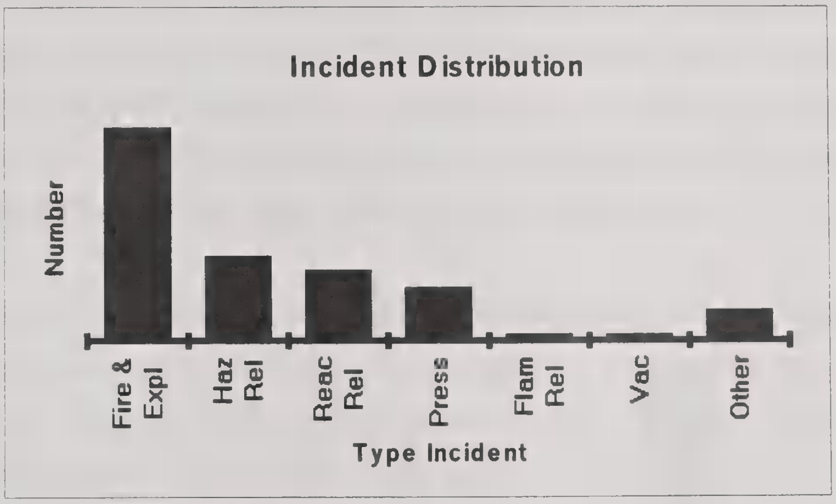

Figure 1. Incident Distribution by Type

Fires and explosions are combined because the distinction between the two is sometimes hard to make. Though "Release of Reactive Materials" is the third bar on this Pareto, it presents an interesting and fruitful example of how we used incident data to drive a risk reduction project. What does the data represented by the third bar of the chart in Figure 1 tell us? If all of the reactive releases are analyzed, an interesting picture begins to emerge. We took the data from the third bar on the Pareto chart above and created a new Pareto chart from that data.

The new chart is called a "nested Pareto" (Figure 2). The nested Pareto shows how Reactive Releases were distributed across our diverse product sectors.

Figure 2. Releases by Product Sector

This chart shows us that over 75% of our releases occurred in one product sector. This is, of course, the classic "Pareto Principal" - 80% of our problems are in 20% of our product sectors. So what should we do with this knowledge? Is there more knowledge to be gained? We repeated the process of developing another nested Pareto from the data represented by Product Sector "A" in the chart above. The incidents from that sector were distributed by the "Product Categories" within the sector. The chart shows this further subdivision of the data (Figure 3).

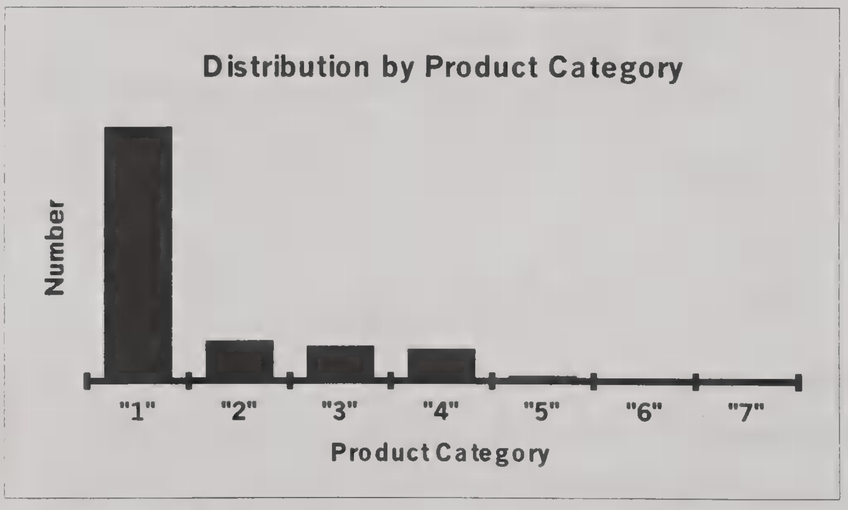

Figure 3. Distribution of Incidents Across Product Categories

This Pareto shows a clearer picture of where our problems were. Product Category "1" is responsible for most of our reactive releases. What did we do with this information? We know <u>where</u> we should act, but as of yet, we don't know <u>how</u> we should act. At this point, further classification of data helped us decide what we should do. The next step was to find out which

chemicals were responsible for our releases. Again we used a nested Pareto (Figure 4) to discover that chemical "A" was the chemical of concern.

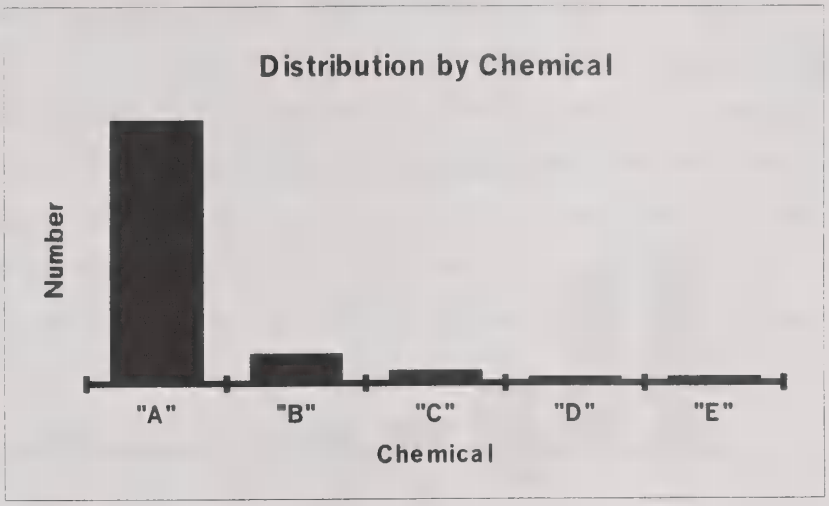

Figure 4. Distribution of Incidents by Chemical

Every time we categorized the data and charted it we learned more. We now knew which product category and which chemical we should focus on. Further, from this information we knew which experts we needed to help us. We also had the information to show those resources why this effort was important to them. These pictures did, literally, say a thousand words. The charts were extremely powerful tools in convincing stakeholders, including upper management and engineering organizations, to dedicate resources to risk reduction efforts.

At this point we went to the leadership in Product Category "1" and presented this analysis, much the way we have presented it in this paper. We requested and were granted the formation of a task force made up of engineering, operations, maintenance and Process Safety personnel. The team had the specific goal of reducing releases of chemical "A".

The formation of the team was a milestone in our efforts to reduce reactive releases. Our analysis of incident data had given us specific direction. We knew which chemical and processes to concentrate on. It gave us an effective presentation tool to convince management to provide us resources. However, we still did not know exactly what we needed to do. This was the work of the task force.

The task force used the same method as we have used up to this point. Review the data, categorize the data, and chart the data. This is a simple concept, but this is not a trivial task. The task force reviewed all of the incident reports of concern and tried to decide how to categorize the different characteristics of the incidents. Several agreements were made on how to categorize data. However, when the data was charted on a Pareto, no clear 80/20 relationship would show

up. When this occurred, we would go back and ask ourselves if there was another way to categorize this data. Ultimately we agreed on the categories shown on the chart below. Since all processes using this chemical were essentially the same design, categorizing the incident data by process components (Figure 5) was a successful strategy for us.

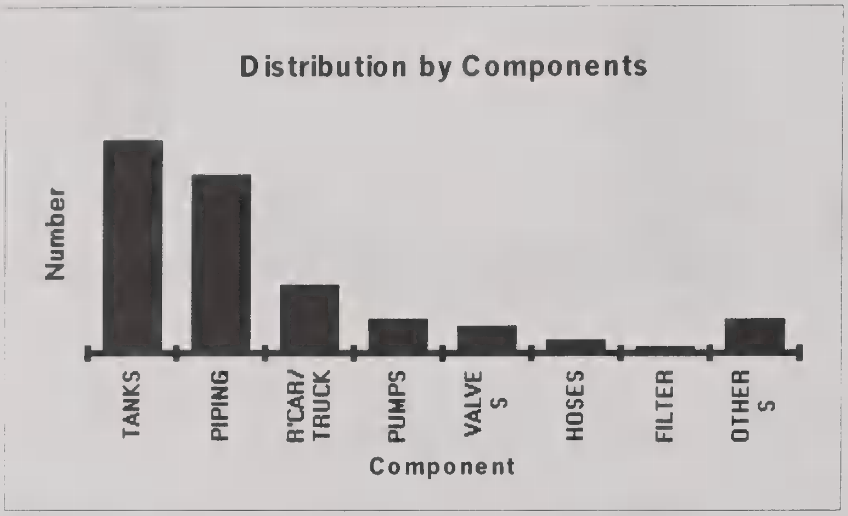

Figure 5. Distribution of Incidents by Component

We were beginning to zero in on the "what" we needed to do. The data revealed that we had a basic, systemic weakness in either the design or operation of our storage tanks and our piping systems. We further broke this data down and found that over 60% of our tank problems were simple tank over fillings (Figure 6).

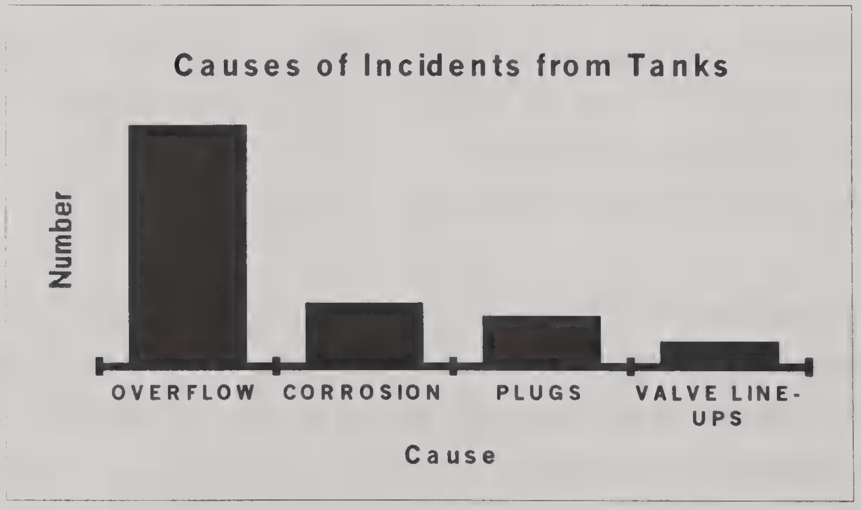

Figure 6. Tank Incidents by Cause

Now we knew <u>what</u> we needed to do. We needed to stop trying to put two gallons of material in a one gallon bucket. The simple answer, of course, was to make a elementary design change and add a high level switch on the storage tanks. The switch was interlocked to the unloading pump and shut off the pump when the high level switch was made. When the tank is being filled, an input to the unloading pump logic is "High Level Switch Not Made". In retrospect, this seems as if

a switch and interlock should have been a basic design feature of our unloading and storage system. But the fact was, a number of processes worldwide did not have this feature. Those without the feature were the source our overflow releases.

Analysis of the piping system failures yielded similar, fruitful information. We found that 70% of our failures were leaks from flanges and the remaining failures were due to corrosion. Further investigation showed that two thirds of our flange leaks occurred in the piping systems from the storage tank to the process. This piping conveys chemical "A" at relatively high pressure (>250 psig) compared to the unloading piping (<20 psig). So we would expect a greater propensity for flange leaks in the higher pressure piping. Similarly, all of our corrosion failures occurred in the low pressure piping from the unloading station to the storage tanks. Since chemical "A" is a corrosive, the procedure of connecting and disconnecting to railcars or trucks provided the perfect opportunity for moisture laden air to contaminate the piping. Again, the data had shown us what we might expect.

For what ever reason, years of Process Safety programs, with qualified engineers at each site and audits conducted biannually, had not revealed these facts to us. Only the analysis of data from our incident database showed us these critical pieces of information. The thrust of our risk reduction effort was focused on tank overflows and increasing the integrity of our piping systems. We began system improvements in 1992.

The results our efforts are shown in the run chart in Figure 7. Prior to our risk reduction effort, we were suffering frequent releases of chemical "A". The range of our release numbers were as large as twice the mean. Since implementation of the task force improvements, our mean number of incidents has dropped by a factor of four. We are suffering only 25% of the releases we experienced prior to our risk reduction effort. Using the incident database and Pareto analysis, we have implemented changes which have reduced our incident frequency by 75%.

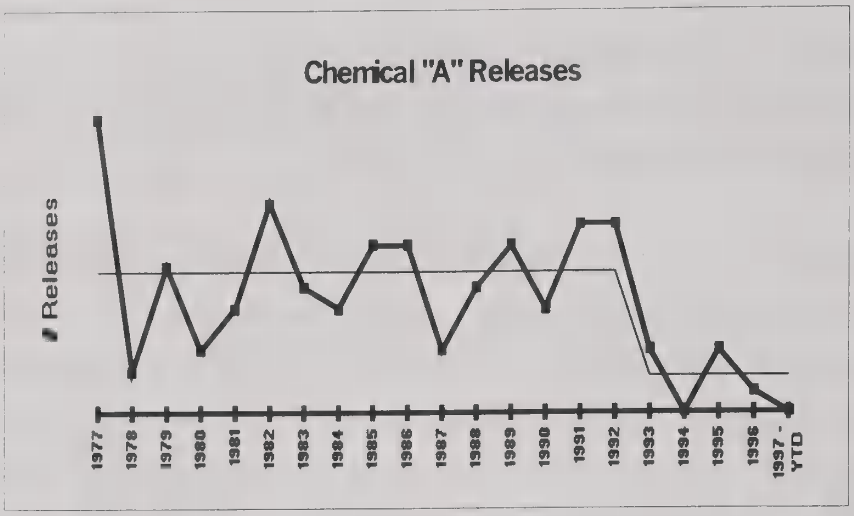

Figure 7. Releases of Chemical "A"; 1977 - 1997 YTD

Though it will take several more years of data gathering to assure that the reductions shown on the chart above are not attributable to random variation, we are confident our initial results will continue.

Case Study 2: Risk Reduction – Process Heaters

Now we will look at a different case study dealing with Fires and Explosions. This case is interesting because it led us to new discoveries about one of our processes and completely changed our strategy for safe design. Several years ago, we suffered a significant incident in one of our processes involving a process heater used to prepare an agricultural commodity for packaging. Fortunately, no one was injured in the incident, but the equipment suffered significant damage due to an explosion. The results of the incident threatened our production capacity and reduced our flexibility. The impact on the process lasted for weeks as repairs and investigation proceeded. In order to fully understand what had happened, we went back to our incident database and began to evaluate all incidents that had occurred in this or similar processes.

We first looked at the numbers of incidents and categorized the incidents according to the process design in which they occurred. Twenty years of data are reflected in the distribution of incidents across three process designs (Figure 8).

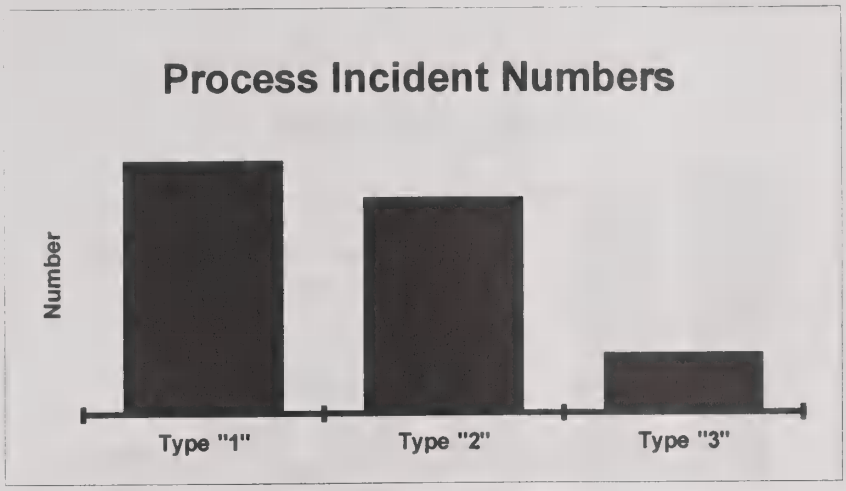

Figure 8. Number of Incidents per Process Type

The chart in Figure 8 taught us that while there was a much higher likelihood of an incident in two of the three types of processes, no clear 80/20 relationship existed with this categorization of incidents. As with the previous case study, we looked for another way to categorize the data. Our second cut at the data looked at the cost of incidents in each of the three types of processes instead of the number of incidents. Incident cost information began to provide us with more revealing information (Figure 9).

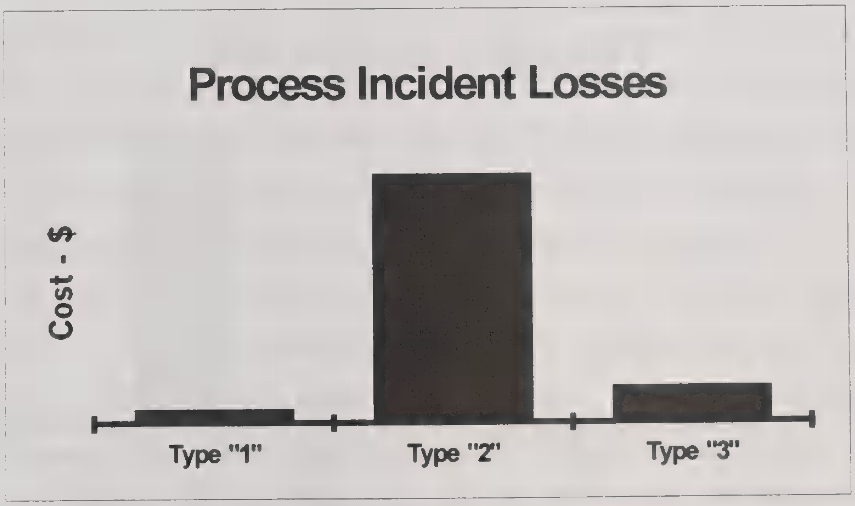

Figure 9. Losses per Incident Type

We now knew our problem was in Type "2" processes. As we had done before, we went to management, obtained resources, and established a risk reduction team. We combed the incident data looking for information which would help us understand the problem more clearly. The key piece of information we found was that though most of the incidents occurred during operation, a number occurred shortly after the process was shut down (i.e., when the gas to the hot air furnace burner was shut off). We again went through the distribution of incident numbers and found that incidents during normal operations outnumbered incidents during shutdown (Figure 10).

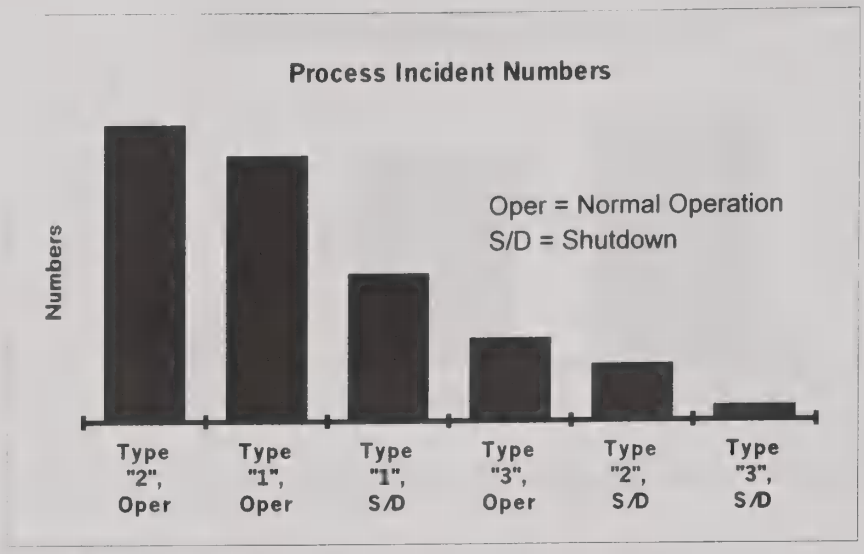

Figure 10. Incidents per Process Type at Various Stages of Operation

The chart in Figure 10 shows a fairly promising "Pareto" relationship. However, the decision to examine cost distribution (Figure 11) provided the real breakthrough for our team. This data was so overwhelming, we really had to understand why the data was so skewed.

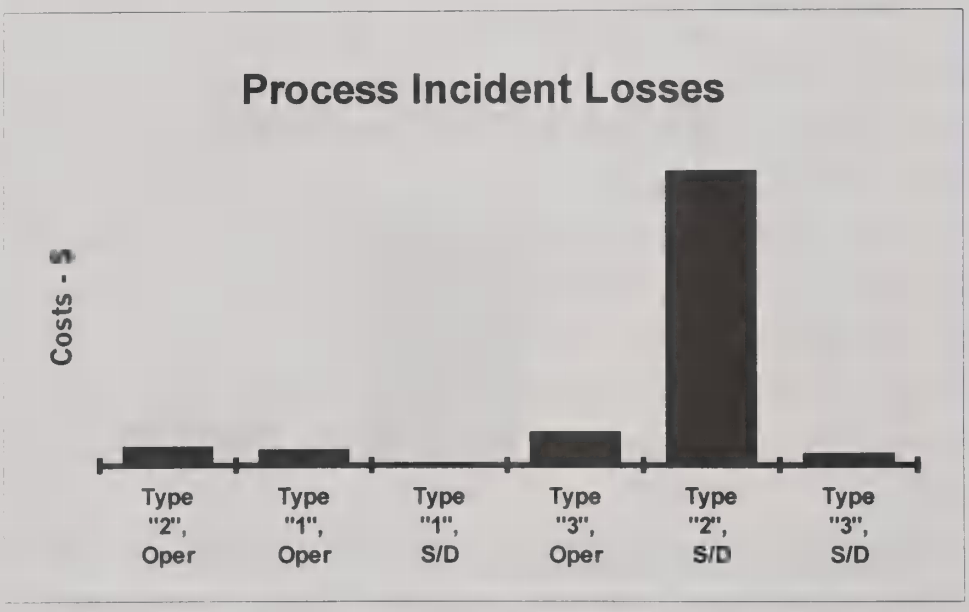

Figure 11. Total Incident Costs per Process Type at Various Stages of Operation

The data shown in Figure 11 initiated a lot of activity from our team, the engineering organization, and the product development organizations. Various theories emerged and were discussed, but no clear hypothesis seemed to explain the data. Finally, process gas analysis revealed that while operating, these processes contained a flammable gas generated by the heating of the agricultural commodity being processed. Further, it was found that during operation the oxygen content in the process gas mixture was only about 12%. A simple schematic of the process is shown in Figure 12.

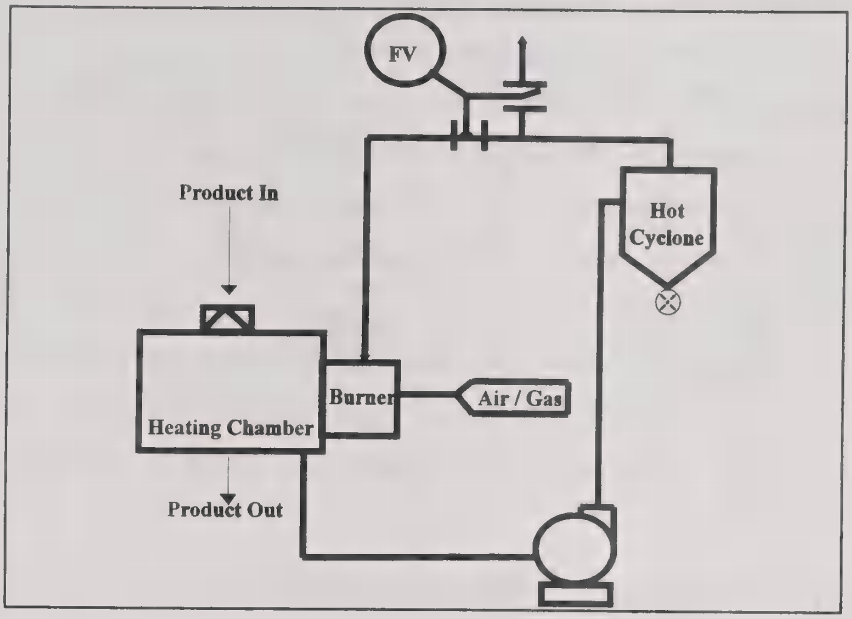

Figure 12. Process Schematic

We now began to investigate our operating procedures and practices. We found that the shutdown sequence simply shut off the gas supply and left the burner combustion air fan running. This allowed fresh air to enter the process gas stream and slowly raise the oxygen level. The process runs at temperatures in the range of 500 to 700° F and there are adequate ignition sources to ignite the flammable gas, if the explosive range is entered. This was our great discovery -- this was a *gas* explosion we were suffering. This was counter to the conventional wisdom for these processes which held that the explosions were *dust* explosions. Across the board, venders supplying the processing equipment had always designed for a dust explosion and provided explosion venting only at the points in the process where dust accumulations were expected (e.g. the cyclones and the heating chamber). The significant damages that we suffered in our incident were due to inadequate venting, and the design bases for the venting of these processes were based on the wrong assumption. From this point on, our strategy was clear. We would redesign our safety features in these processes to minimize the likelihood of an explosion and minimize the consequences of an explosion if one were to occur.

Figure 13 shows a simple diagram of the operating environment to which these processes are subjected. During normal operation, with a depleted oxygen level, the machines operate relatively safely outside of the explosive envelope created by gases generated by heating the product.

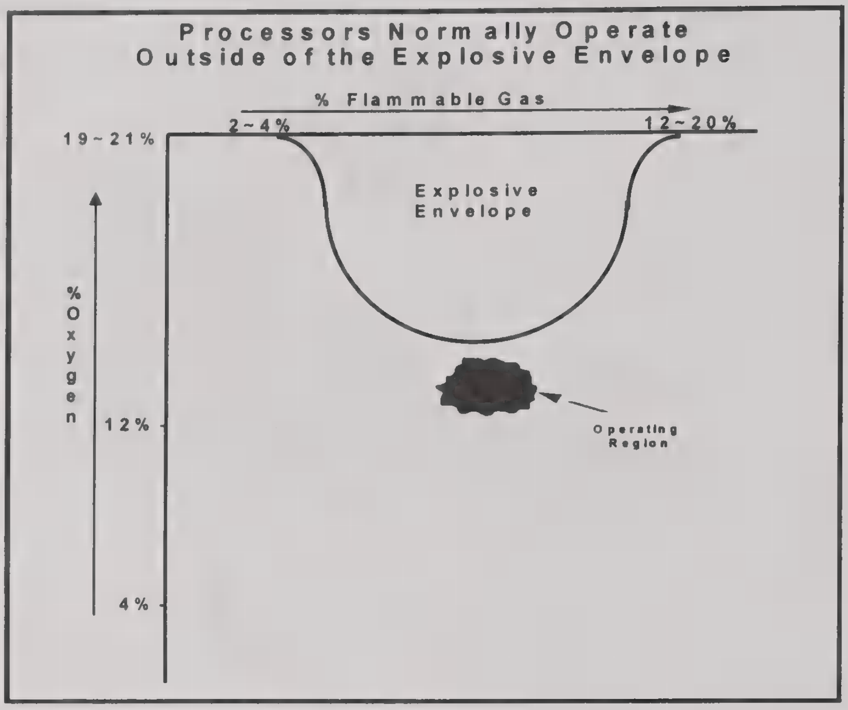

Figure 13. Operating Environment during Normal Operation

When the unit was shut down, the combustion air fan continued to operate. Introduction of fresh air raised the oxygen content and provided the opportunity for the process to enter the explosive range. Our incident data says this will occur with significant consequences about once in every 40 operating years per unit. Figure 14 illustrates this phenomena.

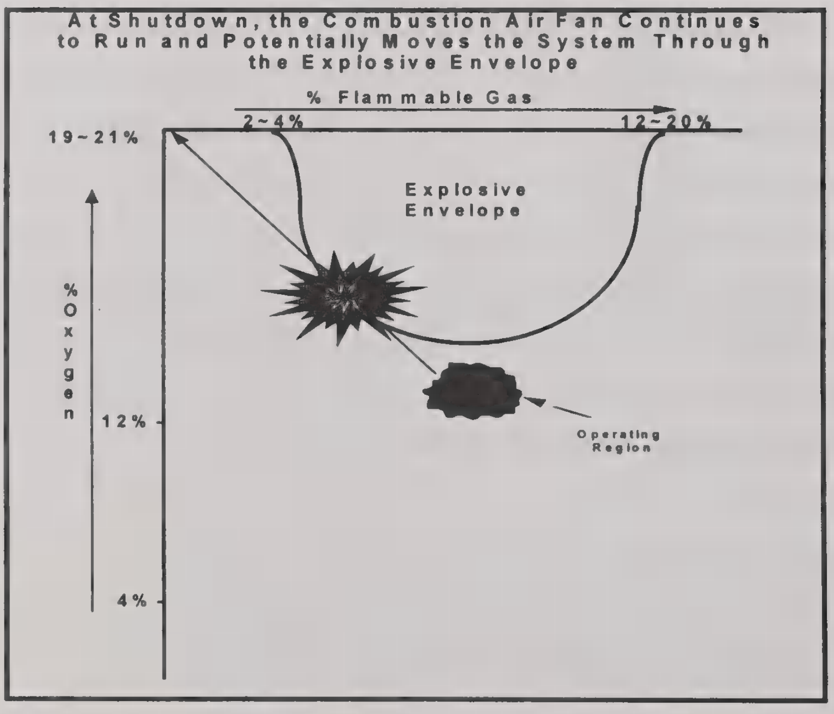

Figure 14. Operating Environment at Shutdown -- Entering Explosive Range

Our first prevention action was to change our shutdown sequence. At shutdown, the gas to the burner is turned off, and the combustion control logics drive the combustion air fan inlet dampers to close. This minimizes the entry of fresh air to the machine. This strategy initially keeps us out of the explosive range. However, we still have an explosive envelope within the process. Our challenge was to figure out how to move from this condition to a safe, complete shutdown. Through research into Bureau of Mines publications on flammable gases, we learned that if we applied a cooling water mist to the process, we could narrow the explosive envelope by lowering temperature, and shrink the envelope by inerting the gas mixture. Cooling water mist gave us the ability to do both. Cooling water rapidly reduced the process temperature, and the resulting steam provided an inerting effect on the process. Figure 15 shows the effect of cooling water mist on the explosive envelope.

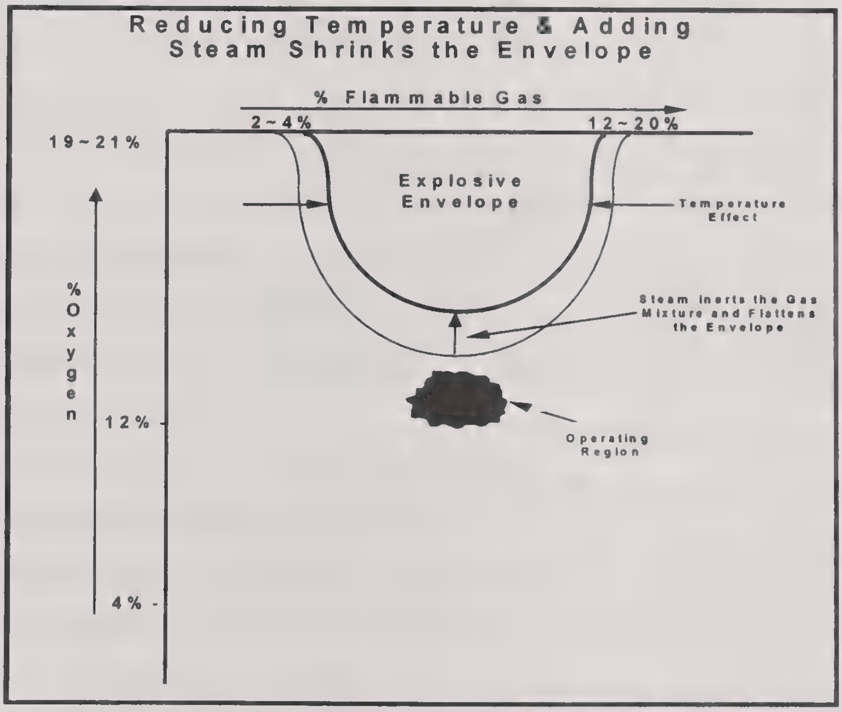

Figure 15. Effect of Cooling Mist on Process Temperature and Explosive Range

We established a desired cool down temperature set point that, when met, would stop water flow to the cooling mist. This cool down set point was well below the temperature where an ignition source could exist within the machine.

Our next step, with the explosive envelope significantly reduced, was to modify the process shutdown logics. The new logics called for the opening of both inlet and outlet dampers to provide rapid purge of our system. Figure 16 shows how we could move to a safe shutdown condition with normal oxygen levels and bypass the explosive envelope. Obviously, this drawing does not show that as we are introducing fresh air into the system, we are simultaneously removing the explosive gasses.

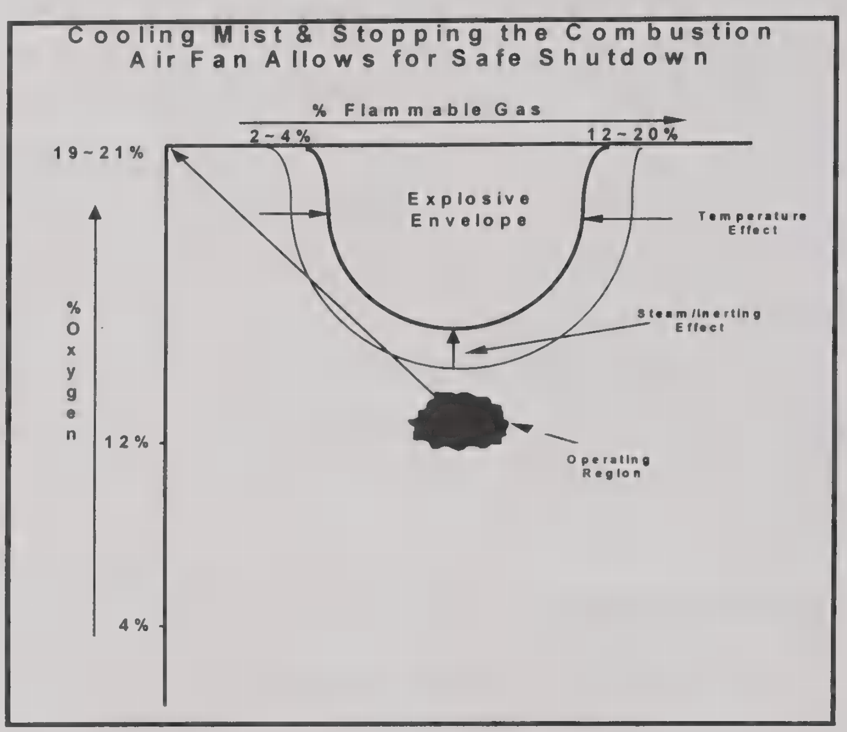

Figure 16. Achieving Safe Shutdown Conditions

These changes in operating procedures and shut down sequences greatly reduced our likelihood of suffering an explosion. However, we are still concerned that we may not understand our process completely and could not guarantee we would never experience another incident. Our next step was to reduce the severity of an explosion if one were to occur. For those who have dealt with explosion venting, it is often difficult to adequately vent an older processes in an existing building and still meet all of the requirements of NFPA 68 "Venting of Deflagrations". What is especially challenging is venting to a safe location (e.g. outside) with the geometry of the processes and their locations within the building. This is exactly the problem we faced. As mentioned, the explosion was assumed to be a dust explosion with venting located only at possible dust accumulation points. What we had discovered is the explosion was a gas explosion and required venting throughout the entire process. It was literally impossible to vent the machine at all of the required locations and direct those vents to the outside. We began to work with Rembe® GmbH, a German firm which manufactures explosion vents and a unique device called a Q-Rohr® Explosion Suppression device. The device is essentially a large flame arrestor attached to a rupture disc. It absorbs the energy of the explosion and quenches the flames. This looked like a promising solution to our venting dilemma.

The Rembe® device was designed for dust explosion and had never been tested for use with explosive gases. Procter and Gamble entered into a joint agreement with Rembe® to test the Q-Rohr®device using a flammable gas which replicated the gases generated during our processing. We completed those tests in early 1996 and proved the devices effectively performed in a flammable gas environment.

With the completion of these test, we had everything we needed to make a step change in the safety design of these processes. We had identified the problem, developed an effective prevention strategy, and determined the Rembe® devices could be utilized to safely vent these machines where we could not direct the venting to the outside.

This was an exciting project to work through. We learned much about our process, and we have made a real difference in assuring safe operation. It is important to note that the discoveries we made were a result of evaluating data. The existence of a database allowed us to review over 200 incidents in these processes and to categorize the data many different ways. The compelling information of the losses associated with a particular design shortly after process shutdown was the driving force of our investigations. We have left many of the details out, but this was a three year effort and we were assisted by some of the outstanding safety firms in the world. What drove us to go to the lengths we did was the data. We could not have tapped that data without an incident database.

Case Study 3: Strategic Direction -- Hazardous Chemicals

The third case study will illustrate how incident data was used in two different ways to help set strategic direction for hazardous chemical management at Procter and Gamble.

Part One -- Hazardous Chemicals Management Systems

In the early 1980s, Procter and Gamble developed a hazardous chemicals management system to improve safety and reduce risks associated with handling hazardous chemicals. In 1992 the OSHA Process Safety Management rule (PSM) and EPA Risk Management Program (RMP) rule, both of which established regulations for managing hazardous chemicals, were in the final stages of development and nearing implementation. We believed PSM and RMP represented the best practices for handling hazardous chemicals. The P&G hazardous chemicals management system closely paralleled OSHA PSM and RMP Prevention Programs, and applied to all P&G listed hazardous chemicals. The list of P&G hazardous chemicals included over 100 chemicals. Some of the P&G listed hazardous chemicals were covered by PSM and/or RMP (e.g., fuming acids). Others were not specifically covered by either regulation (e.g., caustics). To prepare our manufacturing sites for implementation of the new OSHA and EPA programs, we wanted to

convince the sites that in addition to the legal requirement for implementation, the new management programs would actually improve process safety. Again utilizing incident data, corporate process safety demonstrated that, historically, P&G hazardous systems caused the greatest total losses. The chart in Figure 17 illustrates that 60% of process safety losses occurred in P&G hazardous systems.

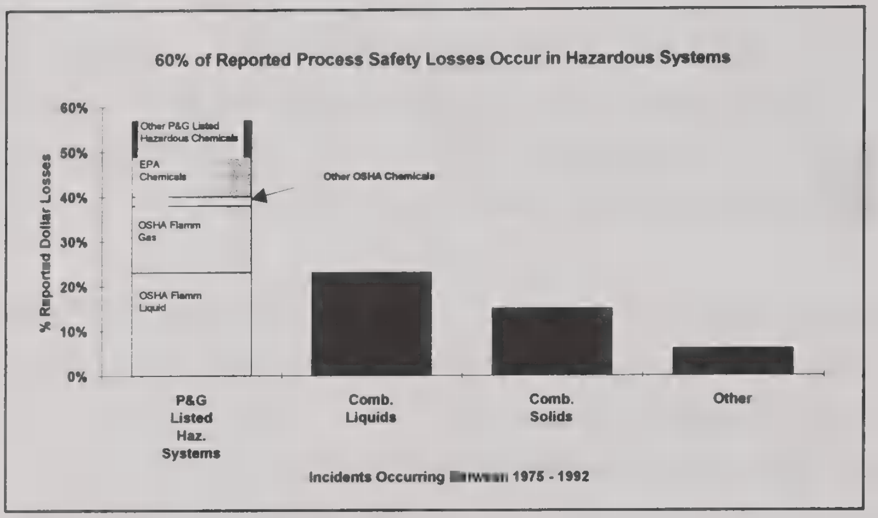

Figure 17. Percentage of Losses for Various Classes of Chemicals

This data helped convince our sites that these chemical management systems had merit and would help improve safety. Implementation of these management systems across all processes handling P&G listed hazardous chemical was the next step. However, closer examination of the data resulted in an unexpected learning.

Further subdivision of the process safety losses by category -- OSHA listed chemicals, EPA listed chemicals, and "other" P&G listed hazardous chemicals -- revealed that 50% of process safety losses were attributable to the first two categories. The third category, other P&G listed hazardous chemicals (over 80), contributed to less than 10% of total process safety losses. Clearly, not all P&G hazardous systems posed the same level of risk. Did all P&G hazardous systems require identical risk management programs? How should this group of "other" chemicals be managed? Relaxing process safety management requirements for these "other" chemicals was fundamentally a different idea for the risk management organizations. This rarely, if ever, happened. As risk managers, we were good at asking for more. What we did not do so well is determine how to eliminate non value-added work. However, we were learning that managing all systems with the same set of requirements diluted process safety efforts and resources. We realized our hazardous chemicals management systems needed to be commensurate with the level of risk. The solution was the creation of a "tiered" hazardous chemicals management system, based on the level of risk posed by the particular process or

chemical. This was a move away from "one-size-fits-all" risk management. Procter and Gamble now categorizes systems according to the level of risk, based on the chemical properties and quantities present in the system. The number of chemicals managed with the most stringent requirements -- now known as P&G Class 1 chemicals -- dropped from more than 100 to less than 20. This focuses process safety resources on the higher risk systems.

Part Two – Flammable Liquids Handling Practices

Another key learning evolved from this same incident data. Systems handling flammable liquids and gases were responsible for most process safety losses, as shown in Figure 18.

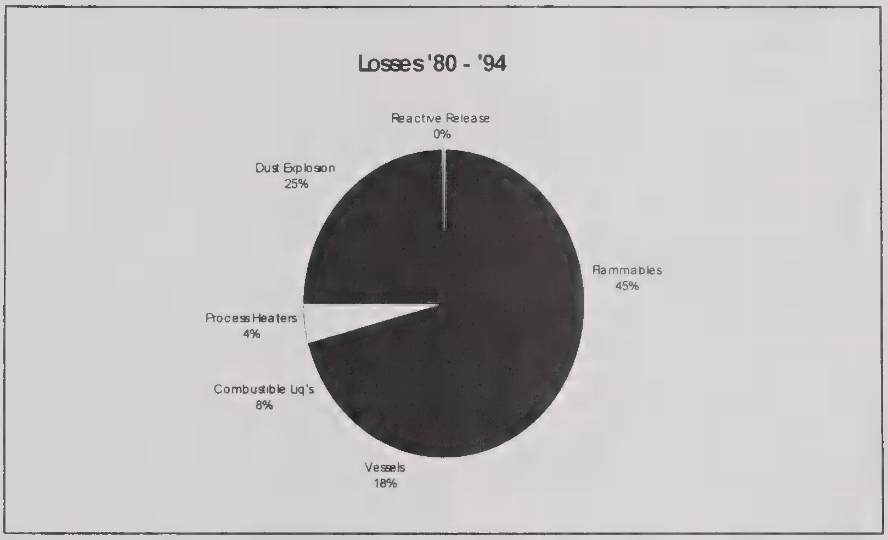

Figure 18. Process Safety Losses by Material Type; 1980 - 1994

Why were these systems suffering disproportionate process safety losses? Were company process safety practices effective? Procter and Gamble process safety practices define company requirements for system design and operation. The practices are based on recognized industry standards, such as NFPA, API, and ASME codes, and company experience. A review of the practices for flammable liquids revealed some weaknesses. From a technical standpoint, the flammable liquids practices were written correctly and aligned with codes and current industry best practices, but they needed to be updated to reflect learnings from recent process safety incidents. The real weakness was how the information was communicated. Comments from engineers and plant personnel using the practices indicated there were too many options, and it was difficult to understand exactly what needed to be done. This decreased the overall effectiveness of the practices and resulted in the practices not being fully implemented at every site. Clear delineation of the requirements would improve system design and decrease the frequency of incidents in systems handling flammable liquids. This was clearly an opportunity for a strategic risk reduction effort -- update the practices for flammable liquids to reflect learnings from recent incidents and

make them more understandable and "user friendly." A major improvement to the practices was the addition of design checklists. These checklists can be used for both design bases and for assessing compliance of existing facilities and systems. The revised practices were issued in late 1996. Will the number of incidents involving flammable liquids handling systems decrease? As of today, we do not have enough post-revision data to make any assessment of the impact of these changes. However, we have chosen flammable liquids as a focus area for risk reduction efforts, and we will use incident data to track results and evaluate the effectiveness of the practices. We believe properly designed systems reduce overall risk. Use of the design checklists should lead to more consistent application of the design requirements, and hence, properly designed systems.

Conclusion

These Case Histories illustrate how much can be learned from process safety incidents and how powerful this information can be. As stated before, many of Procter and Gamble practices and operating procedures are based on company experience. The incident database provided a tool sorting and analyzing information from over 20 years of incident history. Without a database, analysis of this number of incidents would have been much more difficult, if not impossible. The incident data revealed process safety trends and pointed to opportunities for improvement. This allowed corporate process safety to eliminate non value-added work and focus on risk reduction efforts which would have the greatest impact.

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" Tools for Improving Profits"

"It's obvious that *workplace safety* and *responsible care of the environment* is becoming a *competitive advantage* in business. Corporations must constantly develop new initiatives to address risk. They must develop programs that substantially reduce costs so they can get their products to market more competitively, and as Their company grows and expands, provide specialized solutions that improve their company's safety practices and environmental position world wide."

Annual Risk Management Forum, 1996 -- Liberty Mutual Group

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TODAY'S BUSINESS SUCCESS IS A PARTIAL FUNCTION OF:

- RAPID ACCESSING OF COMPLEX INFORMATION
- REDUCING RISKS AND LIABILITIES
- IMPROVING OPERATIONS , PRODUCTIVITY, AND QUALITY
- MEETING OR EXCEEDING REGULATORY COMPLIANCE

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REGULATIONS IMPACTING BUSINESS TODAY:

- Emergency Planning and Community "RIGHT-to KNOW" Act of 1986 (EPCRA)
- ✓ OSHA's Process Safety Management (PSM) Standard 29 CFR 1910.119
- EPA's Risk Management Program (RMP) under section 112(r) of the Clean Air Act
- OSHA's new Ergonomics Program (EPS) Standard 29 CFR 1910.900

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(OSHA 29 CFR 1910.119)

PROCESS SAFETY
MANAGEMENT
OF
HIGHLY
HAZARDOUS
CHEMICALS

"A Tool for Improving Profits"

Purpose of Process Safety Management:

Prevention or **minimization** of consequences of catastrophic release of hazardous, toxic, flammable, or explosive chemicals into the work place or surrounding community.

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What does a Process Safety Management Program Require ?

- Applicability
- Employee Participation
- Process Safety Information
- Process Hazard Analysis
- Operating Procedures
- Training
- Contractor Qualification
- Pre-startup Safety Review
- Mechanical Integrity
- Hot Work Permits
- Management of Change
- Incident Investigation
- Emergency Planning & Response
- Compliance Auditing
- Trade Secrets

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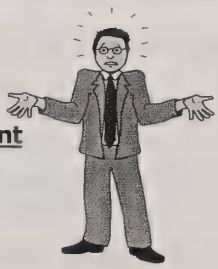
Why is
Process Safety Management
Important?

Why bother with it?



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REGULATORY REASONS:

--- ELEMENTS MOST OFTEN FOUND DEFICIENT BY OSHA

- PROCESS SAFETY INFORMATION
- PROCESS HAZARD ANALYSIS
- OPERATING PROCEDURES
- MECHANICAL INTEGRITY

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ECONOMIC REASONS:

Process Safety Management efforts are not, in a true sense, expenses that represent a burden on a business.

" They represent expenditures designed to lower the overall cost of operations. When wisely spent, such expenditures, in fact, do reduce operating costs."

Process Safety Management efforts have been demonstrated to be

-- Good Business Sense !!!

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Key Elements of OSHA's PSM Standard:

- Employee Participation (YOU)
- *PROCESS SAFETY INFORMATION*
- *PROCESS HAZARD ANALYSIS*
- *OPERATING PROCEDURES*
- Training
- Contractors
- Pre-Start-up Safety Review
- *MECHANICAL INTEGRITY*
- *MANAGEMENT of CHANGE*

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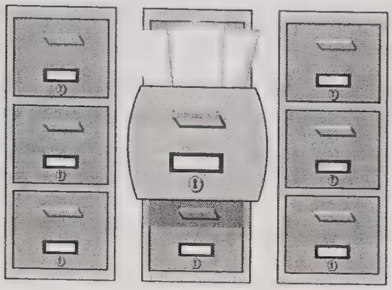
Process Safety Information
29 CFR 1910.119 (d)



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The Purpose of Process Safety Information (PSI)

"Is to compile written process information which will enable the employer and the employees involved in operating a covered process involving highly hazardous chemicals to identify and understand the hazards posed by that process."

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Process Safety Information

- Written information explaining the process hazards.
- Employees must be provided access to this information.
- This information should include;
  - Hazards pertaining to the chemicals used in the process (NOTE: MSDS may contains this information).
  - Data pertaining to the process technology.
  - Data pertaining to the equipment used in the process.

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PROCESS HAZARD ANALYSIS
29 CFR 1910.119 (e)

PROCESS HAZARDS ANALYSIS

The Pinnacle of a Sound Process Safety Management Program

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A PHA is defined as:

- A systematic effort designed to identify and analyze hazards associated with the processing or handling of highly hazardous materials; and
- A method to provide information which will help workers and employers in making decisions that will improve safety.

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A PHA analyzes:

- The potential causes and consequences of fire, explosions and releases of toxic chemicals;
- The equipment, instrumentation, human actions and other factors which might effect the process.

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A PHA attempts to determine:

- The failure points, methods of operations and other factors that can potentially lead to accidents.

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Operating Procedures
29 CFR 1910.119 (f)

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Operating Procedures (Cont'd)

Companies with covered processes must develop and implement operating procedures addressing the following:

- **Steps for each operating phase**
  - Initial start-up
  - Normal operation
  - Temporary operations as needs arise
  - Emergency operations (including shutdowns)
  - Start-ups following outages or shutdowns

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Operating Procedures (cont'd)

- **Operating Limits**
  - Consequences of deviations
  - Steps to correct or avoid deviations
  - Safety systems and their functions

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Operating Procedures (cont'd)

- **Procedures must address Safety and Health considerations such as;**
  - Properties and hazards of process chemicals
  - Exposure precautions including controls (engineering, administrative, PPE)
  - Safety procedures for opening equipment
  - QC for raw materials and inventory levels
  - Control measures in event of exposure
  - Any special or unique hazards

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Operating Procedures (cont'd)

- Operating procedures shall be readily accessible to employees
- Operating procedures shall be current

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Mechanical Integrity
29 CFR 1910.119 (j)

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Mechanical Integrity

- Application :
  - ◈Pressure vessels and storage tanks
  - ◈Piping (including valves)
  - ◈Relief and vent systems and devices
  - ◈Emergency shutdown systems
  - ◈Control (including monitoring devices and sensors), alarms and interlocks

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Mechanical Integrity (cont'd)

- **Written Procedures**:
  - Written procedures must be in place for maintenance
  - Employees must be properly trained
- **Inspection and testing:**
  - Required on process equipment
  - Follow accepted codes, standards and engineering practices
  - Frequency should meet or exceed applicable codes and standards
  - Inspection and tests must be certified

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Mechanical Integrity (cont'd)

- **Equipment deficiencies corrected before further use:**
  - Or in a timely manner (OSHA definition, 6 months or less from date of discovery).
- **Quality Assurance**:
  - Assure equipment meets design specifications
  - Checks and inspections required to assure proper installation
  - Assure maintenance materials, spare parts and equipment meet design specifications

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Management of Change
29 CFR 1910.119 (l)

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The Purpose of Management of Change (MOC) Procedures

"Is to provide a written management system through which contemplated changes to a covered process can be thoroughly evaluated and fully assessed to determine their potential impacts on employee safety and health and to determine needed changes to operating procedures before the changes are implemented."

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MOC Procedures must be written and Implemented for;

- Changes in Process Chemicals;
- Changes in Technology;
- Changes in Equipment; and
- Changes in Operating or Maintenance Procedures

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NOTE:

Replacement of "Like in Kind" is exempted from Management of Change Procedures

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The MOC Procedures must assure that the following considerations are addressed prior to any change:

- The technical basis for the proposed change;
- Impact of change on safety and health of employees;
- Modifications to and updating of operating procedures and process safety information;
- Necessary time period for the change; and
- Authorization requirements for the proposed change.

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MOC also covers Employee notification and training;

" All direct and contract employees involved in operating or maintaining the covered process who's job tasks will be affected by a change in the process must be informed of, and trained in, the change prior to start-up of the process or affected part of the process."

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REMEMBER -- TODAY'S BUSINESS SUCCESS DEPENDS ON:

- MEETING OR EXCEEDING REGULATORY COMPLIANCE
- RAPID ACCESSING OF COMPLEX INFORMATION
- REDUCING RISKS AND LIABILITIES
- IMPROVING OPERATIONS AND PRODUCTIVITY

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Possible Benefits of a Process Safety Management Program for a company:

- Reduced Risks
- Regulatory Compliance
- Reduced Insurance Costs
- Lessened Liability
- Good Public Relations

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Benefits in Operations

- Better Work Environment
- Reduced Overhead
- Increased Productivity
- Use/Manage Resources Responsibly
- Maintain Product Quality
- Maintain Long Term Viability
- Reduced Costs

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Bottom Line Benefit

- Increased Profits

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RECAP --- What does a Process Safety Management Program Require ?

- Applicability
- Employee Participation
- Process Safety Information
- Process Hazard Analysis
- Operating Procedures
- Training
- Contractor Qualification
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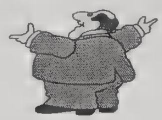

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Modification of Vegetable Oils Using Tung Seed Extracts

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Abstract. Seeds of the tung tree produce a valuable industrial oil enriched in the unusual fatty acid, eleostearic acid. This fatty acid is readily polymerized upon exposure to air, resulting in unique film forming properties that make tung oil a useful component of coatings, inks, and varnishes. Our lab is interested in developing methods to convert low cost vegetable oils into value added drying oils enriched in eleostearic acid. In the current study, we reacted a variety of common fatty acids or triglycerides with crude tung seed extracts to determine the types of products that could be produced. Two different types of reactions were observed. In the first, addition of triglycerides to the tung extract stimulated production of new triglycerides enriched in eleostearic acid. This was likely due to acyl-exchange occurring between the added triglycerides and pre-existing eleostearoyl-triglycerides present in the tung homogenate. In the second reaction, addition of linoleic acid stimulated the production of new eleostearic acid, suggesting that biosynthetic enzymes were present for conversion of linoleic acid into eleostearic acid. To identify the specific enzymes involved in eleostearic acid biosynthesis, fatty acid desaturases expressed during tung oil synthesis were cloned. Current experiments focus on expression of these genes in a microbial system to characterize their activity. This expression system might permit development of a microbial-based, industrial system for production of drying oils from common vegetable oils.

INTRODUCTION

Tung oil, which is pressed from the seeds of dried tung seeds, has been used for hundreds of years as a finishing oil (Fig. 1). The oil dries to a clear, hard, durable film, which explains its widespread usage in coatings applications such as paints, varnishes, lacquers, etc. It is the highest quality drying oil known to man, and is currently used in many industries requiring drying oil applications. Although tung oil was once produced in the United States, the majority of the oil is currently imported from South America and China. The oil is quite expensive, ranging from $1.00 to $1.30 per pound, which is far above a common vegetable oil such as soybean oil, which currently sells for about $0.15 per pound.

Investigation of the fatty acid components of tung oil demonstrated that is largely composed of eleostearic acid (Fig. 1), which accounts for about 80% of the fatty acid composition. Eleostearic acid is an 18 carbon-length fatty acid with three double bonds at the 9, 11, and 13 positions (9 *cis*, 11 *trans*, 13 *trans*-octadecatrienoic acid) (Fig. 1). The alternation of double bonds, referred to as conjugation, renders the fatty acid fairly unstable and subject to oxidation when exposed to air or heat. It is this reactive property of eleostearic acid that leads to polymerization of tung oil when it is spread thin on a surface, and largely determines the unique drying qualities of tung oil.

Our lab is interested in identifying the enzymes involved in the synthesis of tung oil. Identification of the enzymes would open the door to development of methods to convert low-cost vegetable oils into value added drying oils. The most critical enzymes involved in this process would be the fatty acid conjugase, which synthesizes eleostearic acid from the common fatty acid linoleate (2), and the acyltransferases that are involved in moving eleostearic acid from its site of synthesis in the phospholipid fraction to the storage lipid fraction, which is composed primarily of triacylglycerols (Fig. 2). The fatty acid conjugase is likely to be similar to the fatty acid desaturase-2 (FAD2) family of enzymes, which have been shown to be involved in synthesis of the majority of unusual fatty acids found in the oils of plants (1).

To investigate the enzyme activities present in developing tung seeds, crude homogenates were prepared from seeds actively engaged in oil synthesis and reacted with various types of lipids including free fatty acids and triacylglycerols. Results of eleostearic acid biosynthesis and transfer of eleostearic acid to the triacylglycerol fraction are presented. Lastly, the sequences of two tung FAD2-like genes, both expressed in developing tung seeds during synthesis of tung oil, are reported.

MATERIALS AND METHODS

Tung nuts were harvested at various stages of development from the orchards of the American Tung Oil Corporation in Lumberton, MS. Tung nuts, which refer to small apple-sized fruits containing 5 large seeds (Fig. 1), were picked from the trees and seeds were immediately excised and frozen in liquid nitrogen. A portion of the seeds from each fruit were used to determine the oil content, which was used as an indication of fruit maturity.

Homogenates were prepared from tung seeds that were in the early stages of tung oil synthesis. An aliquot of the homogenate was incubated in the presence of trilinolein for 30 min, 2 hr, or 24 hr, then lipids were extracted and analyzed by HPLC. A UV detector was employed to look specifically at those lipids containing eleostearic acid by measuring the absorbance at 271 nm (λ_{max} eleostearic acid). Similar experiments were performed by adding free linoleic acid plus NADH to the homogenate, then extracting and analyzing lipids.

To identify FAD2-like genes expressed in tung seeds during tung oil synthesis, PCR techniques were used to screen a cDNA library prepared from developing tung seeds. Degenerate primers were designed to conserved regions of the FAD2 enzyme family sequences. Two FAD2-like cDNAs were identified, and cDNA fragments spanning the beginning and ends of the FAD2s were obtained using RACE PCR.

RESULTS AND DISCUSSION

The goal of the current experiments was to determine the types of enzyme activities that were present in tung seed homogenates that might be useful for modifying vegetable oils. The reactive ingredient in tung oil is eleostearic acid, and conversion of vegetable oils into an oil enriched in eleostearic acid would allow utilization of the vegetable oils in new processes. Two types of reactions, based on the typical biosynthetic pathway present in developing seeds (Fig. 2), are expected to occur during synthesis of tung oil. One is the biosynthesis of eleostearic acid from linoleic acid, a reaction that is dependent on the cofactor NADH (6) and occurs while linoleic acid is bound to a specific phospholipid called phosphatidyl choline. The second is an acyltransferase-type of reaction (3), in which the newly synthesized eleostearic acid is transferred from phosphatidyl choline to a triacylglycerol, the predominant form of lipid in mature tung oil.

To investigate the synthesis of eleostearic acid, linoleic acid and NADH were added to a tung homogenate and the formation of new eleostearoyl lipids were monitored over time. As shown in Fig. 3, linoleic acid and NADH stimulated the appearance of eleostearic acid, which was not detected in negative control reactions. HPLC analyses indicated that the new eleostearic acid was present as a free fatty acid. This was somewhat surprising, since eleostearoyl is synthesized while bound to phosphatidyl choline. However, it is possible that a lipase reaction occurred to liberate the eleostearic acid from the phospholipid fraction, which is known to occur in the synthesis of other unusual fatty acids, such as ricinoleic acid in castor oil (5).

A similar experiment was performed by incubation of trilinolein with a tung homogenate. Trilinolein, a triglyceride-type molecule, serves as a model compound representative of a vegetable oils. As shown in Fig. 4, trilinolein stimulated the appearance of new triacylglycerols containing eleostearic acid. There was no appearance of free eleostearic acid, as observed before, suggesting that synthesis of new eleostearic acid was not involved in this reaction. These results suggested that trilinolein stimulated an acyl-exchange type of reaction, in which pre-existing eleostearic acid was transferred between triacylglycerol molecules. Such "remodeling" types of reactions have been previously observed in developing seeds (4, 7).

Throughout the course of these studies, a peak was identified by HPLC that was present in early stages of seed development that decreased to very low levels in mature tung oil (Fig. 5). The peak was most abundant at early stages of seed development, then decreased to low but detectable levels while all other eleostearoyl lipids increased dramatically. This pattern is suggestive of a lipid intermediate in tung oil biosynthesis. Purification of the peak, followed by GC analysis of fatty acid composition, confirmed that the lipid contained eleostearic acid. However, no other major fatty acids were observed. Experiments are ongoing to characterize the nature of this lipid. One possibility is that this lipid represents dieleostearoyl-phosphatidyl choline, an expected lipid of newly synthesized eleostearic acid.

To unequivocally identify the tung enzymes responsible for eleostearic acid biosynthesis, we attempted to clone the FAD2-like genes expressed in developing tung seeds. The enzymes responsible for synthesis of the majority of unusual fatty acids in plants, including acetylene, conjugated, epoxygenated, and hydroxylated fatty acids, are most similar to the FAD2 family of fatty acid desaturases (1). The normal FAD2 enzyme is responsible for synthesis of linoleic acid (C18:2) while slightly diverged forms of the enzyme are responsible for synthesis of unusual fatty acids. PCR primers were designed based on conserved regions of the FAD2 enzymes and used to screen a tung seed cDNA library. Two different FAD2 genes were identified that were expressed during tung oil synthesis (Fig. 6). One is likely to be the gene responsible for synthesis of linoleic acid, while the other is responsible for eleostearic acid. Experiments are ongoing to express both of these genes in a microbial expression system to allow synthesis of eleostearic acid in an industrial setting.

CONCLUSIONS

Tung seed homogenates, prepared from seeds that were actively engaged in oil synthesis, contained enzymes capable of modifying fatty acids or triacylglycerols. Two types of reactions were observed: linoleic acid-dependent synthesis of new eleostearic acid, and trilinolein-dependent remodeling of triacylglycerol. The latter reaction likely featured a shuffling of preexisting

eleostearic acid between triacylglycerols, thereby increasing the reactivity of the trilinolein supplied in the reaction. A lipid containing eleostearic acid was identified in developing tung seeds that may serve as an intermediate in tung oil biosynthesis. Identification of this intermediate will provide insight to the pathway leading from site of eleostearic acid synthesis to its final destination in storage oil. Two FAD2 genes were identified that were actively expressed in tung seeds during synthesis of tung oil. Availability of these genes will open the door to development of alternative methods to produce tung-like drying oils from low-cost vegetable oils.

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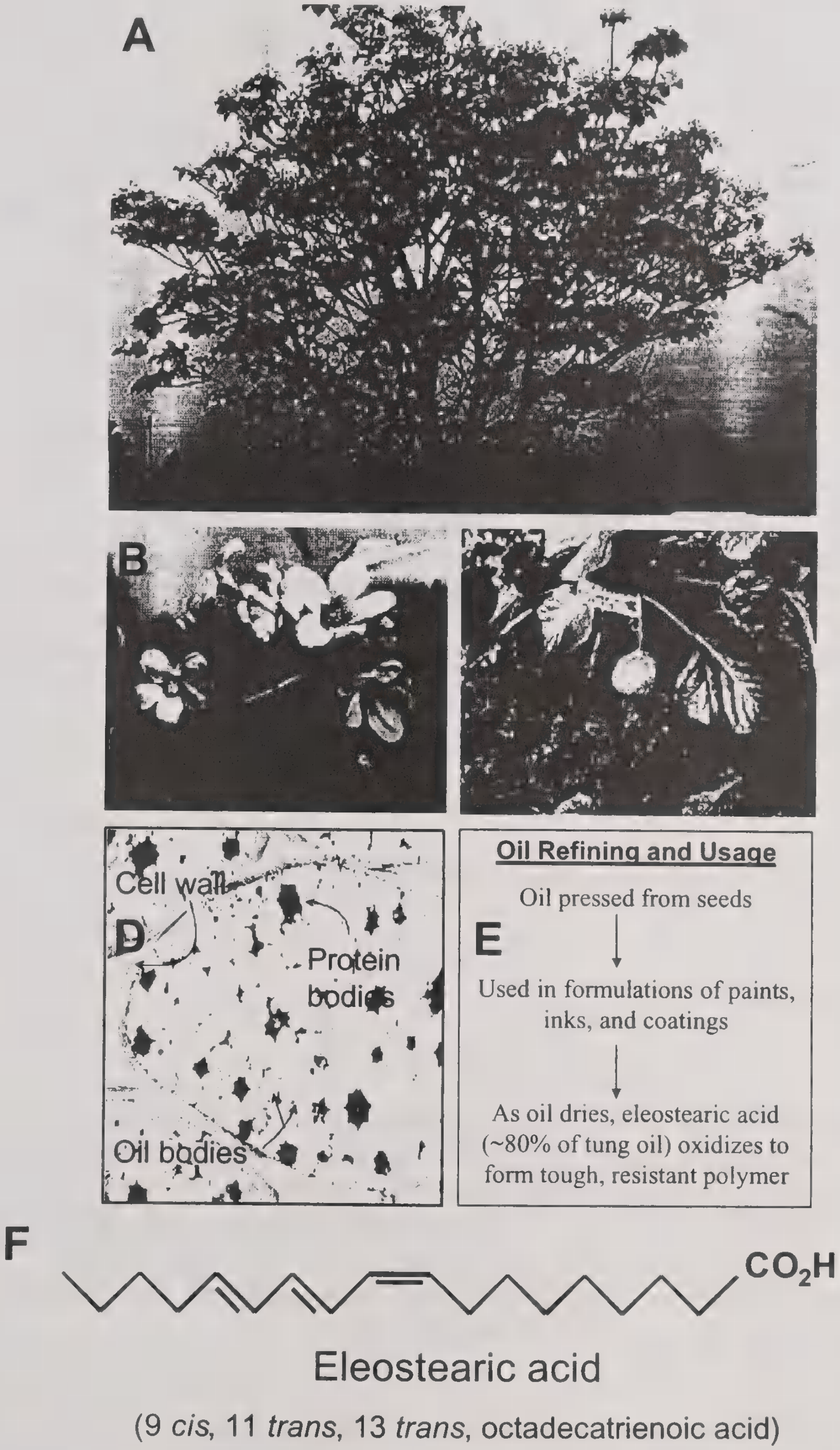


Figure 1. Description of tung tree, fruit, and uses of the oil. (A) Tree. (B) Flower. (C) Fruit. (D) Thin section electron micrograph of tung seed endosperm tissue. (E) Flow chart of tung oil extraction and usage. (F) Cartoon of eleostearic acid.

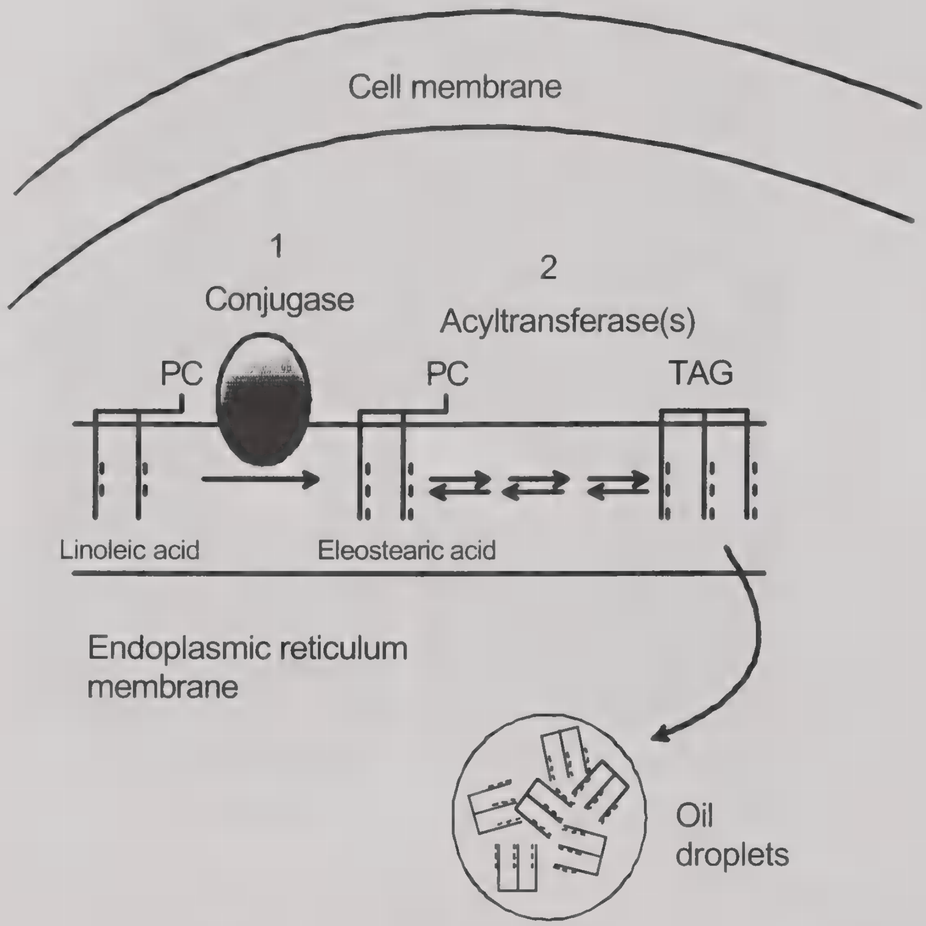
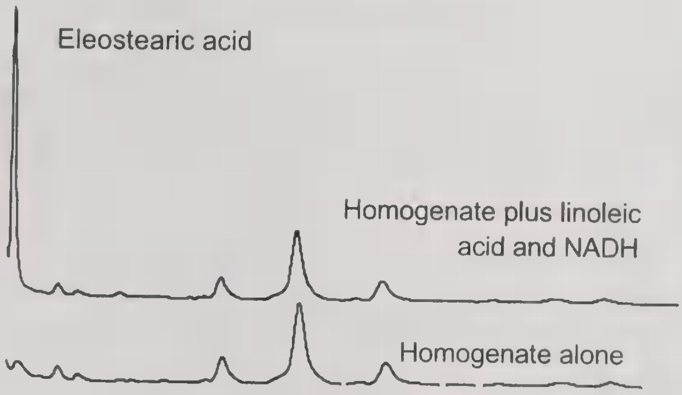
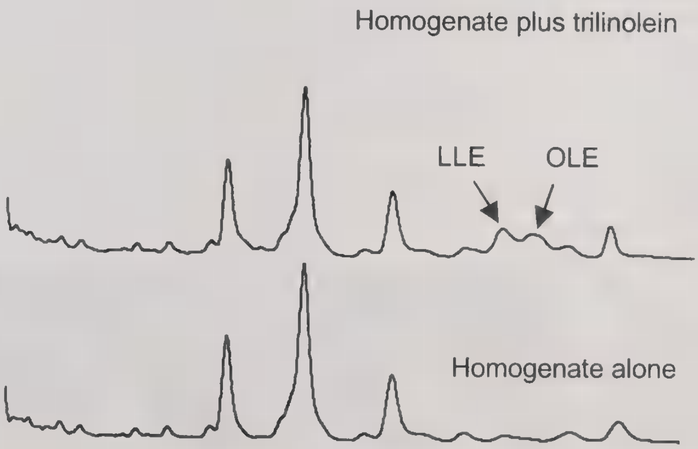


Figure 2. Cartoon of a tung cell actively engaged in tung oil synthesis. Two potential enzyme steps that could be used for modification of vegetable oils are identified: 1. Linoleic acid could be converted to eleostearic acid by the "conjugase" enzyme. 2. Triacylglycerols could be remodeled to increase their eleostearic acid content by an acylexchange reaction involving transfer of pre-existing eleostearic acid with other fatty acids on the vegetable oil. PC - phosphatidyl choline; TAG, triacylglycerol.



HPLC Lipid Profiles

Figure 3. Synthesis of eleostearic acid in a tung nut homogenate. Tung seeds in the early stage of tung oil synthesis were homogenized and incubated in the presence (upper curve) or absence (lower curve) of linoleic acid and NADH. The upper trace shows the appearance of a new eleostearic acid peak in the spectrum. This peak was confirmed to be eleostearic acid by photodiode array (see Fig. 5).



HPLC Lipid Profiles

Figure 4. Production of new triacylglycerols containing eleostearic acid. Tung homogenates were incubated in the presence (upper curve) or absence (lower curve) of Trilinolein. The upper trace shows the appearance of new triacylglycerols containing eleostearic acid. LLE, Triacylglycerol containing 2 linoleic, one eleostearic acids; OLE, Triacylglycerol containing oleic, linoleic, and eleostearic acids.

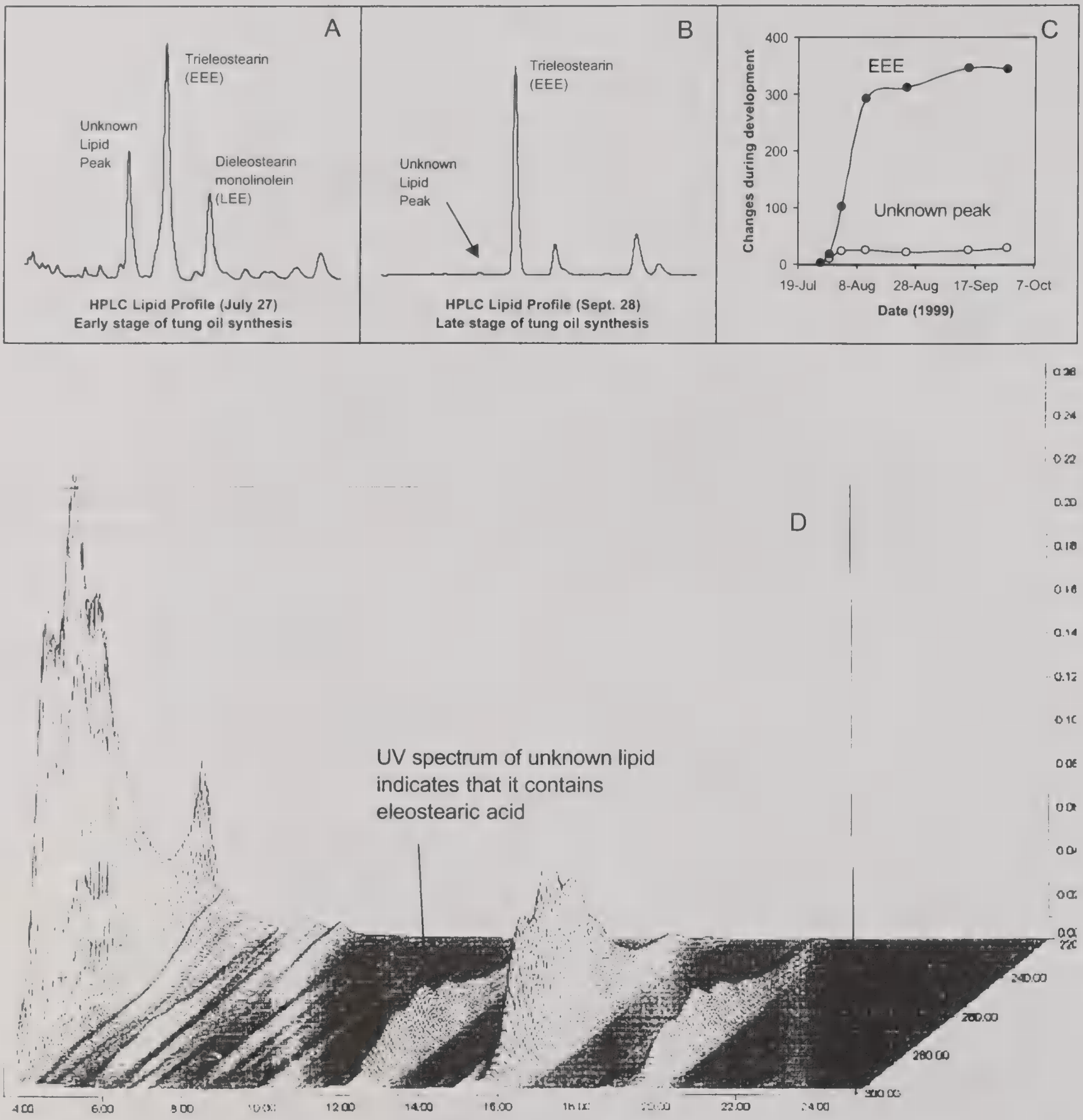


Figure 5. Identification of a prominent lipid species in developing tung oil that contains eleostearic acid. (A) Lipid profile of early-stage tung oil synthesis. (B) Lipid profile of late-stage tung oil synthesis. (C) Changes in relative content of unknown lipid peak and trieleostearin throughout seed development. (D) HPLC lipid profile of early-stage tung oil analyzed using a photodiode array detector. This detector measures UV absorbance spectra, from 220 to 300 nm, as the lipids elute from the column. Any lipids containing eleostearic acid show the UV spectrum typical of this fatty acid (three peaks with absorbance maximum at 271 nm). As shown in (D), the unknown lipid peak contains eleostearic acid. The analysis in (D) was done on the sample shown in Fig. 3 to also prove that the lipid produced upon incubation of the tung homogenate with linoleic and NADH resulted in appearance of new eleostearic acid.

```
                  10        20        30        40        50        60
                   |         |         |         |         |         |
FAD2      MGAGGRMSVPPPP---KKLESEVLKRVPHSKPPFTLGQLKKAIPPHCFQRSVLRSFSYVV
FADX      MGAGGRMSVAPNNSKCEKKESRSVKRVPHTKPPFTLGQLKQAIPSHCFKRSLLRSFSYVV
          *********.*     :* **. :*****:**********:***.***:**:********

                  70        80        90       100       110       120
                   |         |         |         |         |         |
FAD2      YDLTVAFIFYYIATNYFHLLPQPLSYVAWPIYWALQGCVLTGVWVIAHECGHHAFSDYQL
FADX      YDLSLSFIFYSIATTYFHLLPSPITYIAWPVYWAFQGCILTSVWVLGHECGHHAFSEYNW
          ***:::**** ***.******.*::*:***:***:***:**.***:.*********:*:

                 130       140       150       160       170       180
                   |         |         |         |         |         |
FAD2      LDDIVGLVLHSCLLVPYFSWKHSHRRHHSNTASLERDEVFVPKKKSSIRWFSKYLNNPPG
FADX      LDDTIGLILHSSLLVPYFSFKISHRRHHSNIASLERDEVFVPRLKSAIPWYSKYLNNPPG
          *** :**:***.*******:* ******** ***********: **:* *:*********

                 190       200       210       220       230       240
                   |         |         |         |         |         |
FAD2      RLFTLTITLTLGWPLYLAFNVSGRPYDRFACHYDPYGPIYTDRERTEIYISDAGVLAVTF
FADX      RALTLVATLFIGWPLYLAFNVSGRYYDRFACHYDPYSPIYSDRERLQIYISDAMIFVAAY
          * :**. ** :************* ***********.***:**** :****** ::..::

                 250       260       270       280       290       300
                   |         |         |         |         |         |
FAD2      GLYRLAAAKGLAWVICVYGVPLLIVNAFLVMITYLQHTHPSIPHYDSSEWDWLRGALATV
FADX      VLYKIAMAKGLAWLVCIYGVPLLIGNALVVTITSLQHTHVALPHYDSSEWDWLRGGLATV
           **::* ******::*:******* **::* ** ***** ::*************.****

                 310       320       330       340       350       360
                   |         |         |         |         |         |
FAD2      DRDYGILNKVFHNITDTHVAHHLFSTMPHYHAMEATKAIKPILGEYYQFDGTPFYKAMWR
FADX      DRDYGVFNKIFHNATDTHVIHHLFSSMPHYHGVEATRAIKPILGDYYLFDDTPIHVALWR
          *****::**:*** ***** *****:*****.:***:*******:** **.**:: *:**

                 370       380
                   |         |
FAD2      EAKECIYVEADDGDESKGVYWYNKKF
FADX      EAKECLFVEPDEGDNNNGVXWYSNKX
          *****::**.*:**:.:** **.:*
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Figure 6. Amino acid alignment of tung FAD2 and FADX (putative conjugase) enzymes. The cDNAs for each enzyme were obtained from a cDNA library using PCR. The encoded enzyme amino acid sequences are 72% identical, 92% similar. The FAD2 enzyme converts oleic acid to linoleic acid, and the FADX enzyme is expected to convert linoleic acid to eleostearic acid. The genes are currently being expressed in yeast to confirm enzyme activity.

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Oilseed and Grain Processing at the Southern Regional Research Center

Robert J. Hron, Sr. and Michael K. Dowd
Southern Regional Research Center
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Oilseed and Grain Processing at the Southern Regional Research Center

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INTRODUCTION

In 1940, the Agricultural Research Service (ARS) opened four major agricultural research centers in the East, Midwest, South and West regions of the US. The mission of these centers was to develop new uses for local agricultural products and byproducts and thereby provide a larger monetary return to the American farmer. Over the years, many products and processes have been developed within the Centers. A few of the noteworthy accomplishments include improved production of penicillin, development of instant mash potatoes, development of durable-press treatments for cotton fabrics, and methods to minimize the production of the fungal metabolite aflatoxin. Pilot-plant facilities were included in each Center. However, after 60 years the existing facilities need to be modernized if they are to keep pace with new technology. These renovations were started in 1997 and will be completed in the next five years.

At the Southern Regional Research Center, the Commodity Utilization and Food Processing and Sensory Quality Research Units will oversee the non-fiber pilot-plant facilities. The Commodity Utilization facilities will be used primarily for oilseed and grain processing research with a concentrated effort on solvent extraction. These facilities are divided into two separate two-story areas. One plant, with approximately 2,400 ft^2 of space, will be explosion proof and used in various solvent extraction and oil/meal recovery operations. The second plant will have approximately 2,700 ft^2 of space and will be used for operations not requiring explosion-proof facilities, which will include oilseed and grain preparation operations. When completed in the fall of 2001, these plants will be ARS's principle facility for oilseed processing. The pilot-plant housed within the Food Processing and Sensory Quality Research Unit will focus on food formulation issues but will also have some equipment that will be useful for oil and protein research. The equipment available in these pilot-plants, their characteristics, and a review of some recently completed projects is described below.

EQUIPMENT SUMMARY

Extraction. The centerpiece of the solvent pilot-plant is a French Oil Mill Machinery Co. batch oil extraction system. This is a complete solvent extraction plant, which can operate in deep (72") or shallow (30") bed modes. The 1.5 ft^3 extraction column handles batches of between 5 and 100 lbs., depending upon feed product density and operating mode. The plant also includes miscella and rinse storage tanks, a miscella evaporator-stripper, and a spent material desolventizer-toaster. The plant produces solvent free crude oil and defatted meal.

The solvent lab also has several cold-solvent, basket-type batch extractors that can handle a small quantity (1-100 lb.) of fine or coarse materials. This unit includes 12, 2 ft^3 stainless steel baskets. For small scale extractions (<500 g), jacketed, stainless steel extractors are available that accommodate solvent flow.

In addition, an explosion-proof liquid-liquid extraction unit is housed within the pilot-plant. This unit is designed for countercurrent contacting of the feed material with a stripping solvent. The assembly has 36 stages and has solvent recovery elements.

Evaporation. The laboratory is well equipped with evaporation equipment that can be used for solvent stripping. The French extraction unit contains its own dedicated solvent recovery unit. Additional evaporators include both rising and falling film instruments. The plant also houses an Artisan Rototherm thin-film evaporator and oil stripper.

Specialized solvent operations. A number of solvent-related projects have required specialized equipment. Consequently, a number of auxiliary pieces of equipment are also operational. These include a ~5 ft^2 explosion-proof vacuum crock slurry filter, a set of 1 ft^3 Sparkler filters, a Morehouse stone mill (useful for grinding solvent slurries), a stainless steel variable speed De Laval oil refining centrifuge, and a liquid cyclone module capable of handling solvent slurries. The facility also houses a Patterson Kelly vacuum/pressure reactor and a Groen vacuum/pressure reactor. Of a less specialized nature are explosion-proof ribbon blenders, a rolling drum blender, and a number of laboratory-scale V-blenders.

Drying. Drying facilities are essential to most agricultural-food processes, and the pilot-plants are well equipped to handle these operations. The various laboratories have a continuous gas-fired belt-type air dryer, a Proctor and Swartz forced-draft oven capable of operating to 200 °F, a pilot-plant scale Patterson Kelly explosion proof "V"-type solids processor, which can be used for mixing as well as drying and desolventizing operations, and a force-convection roaster, which can be operated in up or down draft modes. Specialized drying equipment include a large (~1000 lb/hr) microwave vacuum dryer and a 25-L Vertis freeze dryer with temperature controllable shelves.

Milling. For particle attrition operations, the laboratory has Alpine Contraplex and Kolloplex fine grind pin mills, two 8” Bauer mills with various grinding plates, a Reitz hammer mill, a Fitzpatric hammer/knife mill, a pilot-plant Wiley mill, and a 1-gal scale ball mill. For cracking and flaking operations, a set of 24” Ferrell-Ross shearing flaking rolls and a set of 12” Allis Chalmers cracking and flaking rolls are available. Laboratory mills include a Retsch pulverizing mill, Spex Certiprep ball and liquid nitrogen attrition mills, Wiley laboratory and intermediate mills, two Straub (Quaker City) mills, and a GlenMills micro hammer-cutter mill.

Extrusion. Extrusion operations are conducted with a 4.5” diameter, 300 lb/hr, variable-speed Anderson International extruder/expander with an oil cage or an Anderson International DuoExpeller. For operating on a smaller scale, a 30 mm Werner and Pfleiderer twinscrew extruder and an experimental computer-controlled 18 mm Leistritz

twinscrew extruder are available. The Center also operates a pilot Brabender extruder and a Dakota laboratory expeller.

Classification. The facilities contains a Zig-Zag pilot-plant scale air classifier and a Masac AcuCut classifier. Sweco and Kason vibrating screen particle separators, which can be run either wet or dry in continuous or batch modes, are also available.

APPLICATIONS

The French oil plant has been used for recovering crude oil from cottonseed, soybeans, and rice bran (1). In principle, the plant can be used for testing the extraction efficiency of different solvents and experiments are in development to use the plant for this purpose. The cold solvent basket system has been used for oil extraction of cottonseed, soybeans, oats, rice bran, corn fiber, and wheat germ. This system has proved to be very convenient for testing solvents, e.g. acetone and alcohols (2-5). The expeller and expander are used frequently for seed preparation prior to oil extraction.

A large number of seed milling operations are performed routinely in the plant. These include milling of cottonseed, rice, and oats for flour with the Alpine Contraplex pin mill (6), dehulling of cottonseed with the Bauer mill, particle size reduction of pecan shells and bagasse with the Fitzpatric hammer/knife mill (7), and the formation of peanut butter with the Morehouse stone mill. The Bauer mills or cracking rolls are used for the hull fracturing of soybeans and peanuts. The separation of hulls from meats or the separation of large amounts of material by particle size is accomplished with the continuous sieve separators. For large-scale separations the Zig Zag air classifer is useful, and for very fine materials, the Masac AcuCut air classifier is used. The Reitz mill has been found to be effective for milling crawfish shells for carotenoid recovery (8).

The vacuum/pressure reactor has been used for ammoniation of cottonseed to eliminate aflatoxin (9) and the chemical retting of kenaf fibers (10). The liquid cyclone process was designed specifically for the recovery of a high-protein, low-gossypol cottonseed product suitable for food applications (6,11). The ribbon blenders are routinely used for adjusting the moisture content of products and for the mixing of ingredients for animal feed formulations.

The drying equipment has been valuable in the production of a wheat substrate for the biological control of aflatoxin producing strains of *Aspergillus flavus* (12) and for testing the usefulness of a microwave pretreatment for the drying of oilseeds (13).

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Antioxidants and Sunscreen Products from Veronia galamensis Epoxidized Triacylglyceride

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Antioxidants and Sunscreen Products From Vernonia galamensis epoxidized triacylglyceride.

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ABSTRACT

Compounds prepared from the naturally epoxidized triacylglyceride of *Vernonia galamensis* seed oil exhibited antioxidant activities when studied by a model reaction of the initiated oxidation of cumene. It was shown that the rate constant of inhibition, k_7, depends on the size and the nature of the radical, $R^{\bullet}$, whose magnitude was found to be

$$k_7 = 10^{7.0 \pm 0.5} e^{\frac{(4200 \pm 700)}{RT}} lmol^{-1} s^{-1}$$

These phenolated vernonia oil derivatives were incorporated chemically to polystyrene and polyurethanes forming polymeric antioxidants useful for master batches for polymeric materials. Further reactions of vernonia oil with sunscreen products gave rise to oils with inherent sunscreen properties capable of use direct as oil cum UV preventing agent in one.

INTRODUCTION

Vernonia galamensis has been shown to be a rich source of vernonia oil, an epoxidized triacylglyceride. This plant is a native of East and Central Africa, which is now under developmental stage as a potential industrial crop. The maximum seed yield has been reported to be 2227 lbs/acre (249.6 gm / m^2 compared with an average yield of 1926 lbs/acre (215.9 g / m^2 for soybean oil in the USA[1].

Vernonia oil has been used in coating formulations[1] has been used as an excellent PVC plasticizer and stabilizer as a reactive diluent for high solid alkyd resins in epoxy formulations and as a good component of interpenetrating polymer networks[2-8)]. Fatty amides[9)], fatty acid bisamides[10)] hydroxy alkoxy fatty esters and dibasic acids[11)] as well as other derivatives have also been synthesized from vernonia oil.

The modern trend has been to tie polymer additives down as it were, in an effort to enhance the properties and performance of commodity polymers[12)] and thus prevent the loss of active components. Since the loss and migration of additives is a cause of many concerns especially in food and medical applications, a binding of the antioxidant to the polymer backbone could produce a superior polymeric material.

The present paper deals with a short preview of the functional properties of a series of phenolated triacylglyceride derivatives (PTAGD) formed by the reaction of vernonia oil with aromatic acids.

The new model compounds prepared from vernonia oil and the well known phenolic antioxidant 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid(DTBH) exhibited antioxidant activities when studied by the model reaction of initiated oxidation of cumene[13,14)]. The phenolated triacylglycerides (PTAG) were copolymerized with styrene and polyurethane to form polymeric antioxidants, DSC study of which indicated their stabilization.

Additional compounds prepared using a known sunscreen product gave rise to oils with inherent sunscreen properties capable of use direct as oil cum UV preventing agent in one. This is in line with the need for sunscreen products requiring the formulation of applications which could not pose a loss after their application on human skin, thus exposing the unsuspecting individual to dangerous UV rays. This seems to be the case in this study with the aim of forming sunscreen products attached directly to oils which can then be used on their own or mixed with lotions which can be applied to hands or the body without the risk of loss of the active ingredients. *In vitro* test methods were employed using a substrate which obviates human test samples [15,16)]

EXPERIMENTAL

Materials and methods

Mass spectrometry was conducted by the use of MAT 711 of Varian Mat. NMR spectra were measured on AC 250 and AM 270 SY Bruker spectrometer, while IR spectra were recorded with a Nicolet FT-IR-Interferometer System 5 SXC.

Compounds used were purified before use. AIBN was purified by double recrystallization from ethanol and benzene. The AIBN was double recrystallized from ethanol and benzene. Irganox 1076 was from Ciba-Geigy.

Trivernolin, divernolin and vernolic acids were obtained from Vernonia galamensis seeds by the method of cold temperature separation.

Antioxidant activity

The antioxidative activity of compounds <u>5</u>-<u>9</u> and polymer <u>14</u> were studied with the use of a model reaction of initiated oxidation of cumene[13,14)]. Oxidation was conducted in the atmosphere. As initiator 2,2'-azobisazobutyronitrile (AIBN) was employed. The kinetics of AIBN decay has been extensively studied [17-21)].The volume of the reaction mixture, which is cumene plus the other ingredients, was 10 ml. The rate of oxidation reaction was defined on oxygen consumption. A volumometer was used for determining the amount of consumed oxygen. The initiation rate is obtained using the expression

$$W_i = ek_d[AIBN] = 1.1x1.58x10^{15} e^{\frac{(-30800)}{RT}} [AIBN]$$

with W_i = the initiation rate,

e = the radical yield (equal to 1.1 for cumene) and

k_d = the rate constant for the initiator decay.

Synthetic method

The reaction of the epoxidized triacylglyceride, diacylglyceride (divernolin) and vernolic acid methyl ester with DTBH were conducted in an atmosphere of nitrogen at temperatures between 120 – 180° C in the presence of a basic ammonium bromide catalyst.. Copolymerization was effected by reacting the ETAG with styrene and heating at 80°C in an inert atmosphere for four hours and then transferred into an oven and heated for 24 h.

Results and discussions

All the obtained compounds and polymers of the nature of sterically hindered phenols exhibited antioxidant activities. This is indicated by the oxygen consumption experiments performed in the initiated oxidation of cumene in the presence of these products.

The following known scheme represents antioxidative activity[23, 24)]

Chain initiation :

$$AIBN \xrightarrow{W_i} 2r^\bullet + RH \longrightarrow R^\bullet + O_2 \longrightarrow RO_2^\bullet \qquad (1)$$

With W_i as the initiation rate.

Chain propagation:

$$RH + RO_2^\bullet + O_2 \xrightarrow{k_3} ROOH + RO_2 \; (2)$$

Chain termination

$$InH + RO_2^\bullet \xrightarrow{k_7} In^\bullet + ROOH \qquad (3)$$

$$In^\bullet + RO_2^\bullet \xrightarrow{k_8} \text{inactive products} \qquad (4)$$

$$In^\bullet + In^\bullet \xrightarrow{k_9} \text{inactive products} \qquad (5)$$

with RH: cumene, $R^\bullet$ and $RO_2^\bullet$: cumyl and cumyl peroxy radicals, ROOH : hydroxyperoxide of cumene, InH: studied products, $In^\bullet$: radical of the inhibitor.

Now, if the above stated scheme is fulfilled, the following relations hold:

$$f_7 = \tau \frac{W_i}{[InH]} \qquad (1)$$

$$k_7 = -\frac{2.3k_3 \lg(1-\frac{t}{\tau})}{\Delta O_2} \qquad (2)$$

and from which it is possible to calculate the parameters of antioxidative activity of compounds 5 to 9 and the polyurethane 14.

Here τ = induction period of oxidation reactions,

f_n = coefficient of inhibition for one molecule of inhibitor,

n = number of inhibiting groups in the molecule, [InH] = concentration of compound,

k_7 and k_3 = rate constants of chain termination and propagation, respectively,

t = the time for reaction and

ΔO_2 = amount of consumed oxygen.

The kinetic curves of oxygen consumption are plotted in Fig 1 and the half-logarithmic graph is shown by Fig 2, from which we calculate the kinetic parameters of antioxidant activities of the inhibitors, with the induction period determined graphically, are all indicative of activities that can be performed by chain propagation inhibitors[23,24)] and thus showing that the synthesized products possess antioxidative properties.

The rate of chain propagation, k_3 for cumene can be calculated from the equation

$$k_3 = 10^{6.7\pm0.3} e^{\frac{(-9800\pm400)}{RT}}$$

From Table 1, it can be observed that the coefficient of inhibition, f_n, decreases with temperature while the rate constant increases.

The synthesized compounds do possess antioxidative properties as indicated by their reaction with cumyl peroxide radicals. By analyzing the results of the experiments we can state that for all phenols studied, the rate constant of inhibition, k_7, depends on the size and the nature of the radical, $R^\bullet$. Further, the unit magnitude of k_7 for all the compounds was found to be

$$k_7 = 10^{7.0\pm0.5} e^{\frac{(4200\pm700)}{RT}} lmol^{-1}s^{-1}$$

From the foregoing, we can say that the synthesis of macromolecular antioxidants fit to be used in the growing field of environmentally friendly polymers has been described based on the naturally epoxidized vegetable oil.

SUNSCREEN PRODUCTS

The products with sunscreen moieties were studied by the use of the stratum corneum *in vitro*. Thin sections were cut and the spectrophotometric studies made before and after their simulated solar irradiation. This method was employed by taking skin samples from 20 different individuals, supplied from hospitals, since the mean protection factor is defined as the arithmetic mean of individual protection factors of 20 test persons. The normal procedure is where individuals are tested by applying the sunscreen product to a given area of the body and exposing it to a UV source for a specific length of time.

The results obtained show a possibility of creating sunscreen-oil compounds which could be employed without risk of much loss of the protective power of the application much like the application of Vaseline on skins during bathing with water repulsion and reduction in the 'charring' effect to the skin. This is true given the hydrophobicity of the resulting sunscreen-cum-oil (SCO) product.

Samples were given to the RP-HPLC and compared by the use of authentic samples to identify the peaks of interest. Use of control was important. For good observation, different wavelengths were taken. The penetration experiments show that the sample from the p-amino benzoic acid and p-methoxycinnamic acid was able to pass through the skin, with the new compounds not seen to penetrate.

Tests were conducted to compare their effectiveness on epidermis after several hours of water immersion to simulate strenuous activities. It was found that the SPF did not alter to any large extend implying a resistance to sunscreen loss due to vigorous activity or water.

UV absorption measurements were routinely performed to assess absorptions in the UV-A or UV-B areas as an indication that the product would absorb the light in this region. Thus a 0.1 % solution of the triacylglyceride sunscreen formulation (TAGSF) dissolved in ethanol or ethyl acetate, depending on the solubility was studied by UV spectrometry.

From Figure 3 it can be observed that the TAGSF shows absorption in the UV- B area and they all taper off from 340 nm. The sunscreen effect can only be seen for the region of UV-B (280-320 nm), an area which needs not reach the skin to protect it from the deleterious effect of the sun's rays. Further studies are being considered for compounds with potential UV-B protection to create a broad spectrum SCO formulation.

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TABLE 1 ANTIOXIDATIVE ACTIVITY OF STERICALLY HINDERED PHENOLS 5 TO 9 AND POLYURETHANE 14 (InH)

InH	5	6	7	8	9	14
Coefficient of Inhibition, *fn*						
40°C	9.3±0.7	4.2±0.2	5.1±0.3	6.7±0.8	3.8±0.2	12±1.4
60°C	8.7±0.6	3.8±0.2	4.1±0.3	2.6±0.2	3.5±0.2	4.4±0.2
80°C	5.4±0.2	2.8±0.1	2.4±0.2	1.2±0.1	2.2±0.1	1.8±0.2
Constant Rate (Reactivity) in $k_7 \times 10^{-4}$ l mol^{-1} s^{-1}						
40°C	1.2±0.1	1.3±0.1	1.5±0.1	0.8±0.1	1.0±0.06	0.7±0.1
60°C	1.7±0.1	2.0±0.1	2.3±0.1	1.5±0.1	1.4±0.1	2.0±0.1
80°C	2.3±0.1	2.5±0.1	2.7±0.1	1.8±0.2	2.1±0.15	2.4±0.2
Activation energy, E, in Kcal mol^{-1}	3.5±0.2	3.6±0.2	3.2±0.2	4.5±0.3	4.1±0.2	6.7±0.6
Preexponent lg A in l mol^{-1} s^{-1}	6.5	6.7	6.4	7.1	6.8	8.6

At 40°C, $W_i = 3.5x10^{-8}$ Lmol^{-1} s^{-1}

At 60°C, $W_i = 3.5x10^{-8}$ Lmol^{-1} s^{-1}

At 80°C, $W_i = 3.5x10^{-8}$ Lmol^{-1} s^{-1}

[InH] $\approx 1.4x10^{-5}$ molL^{-1} to $1.5x10^{-4}$ molL^{-1}

ZEYNALOV

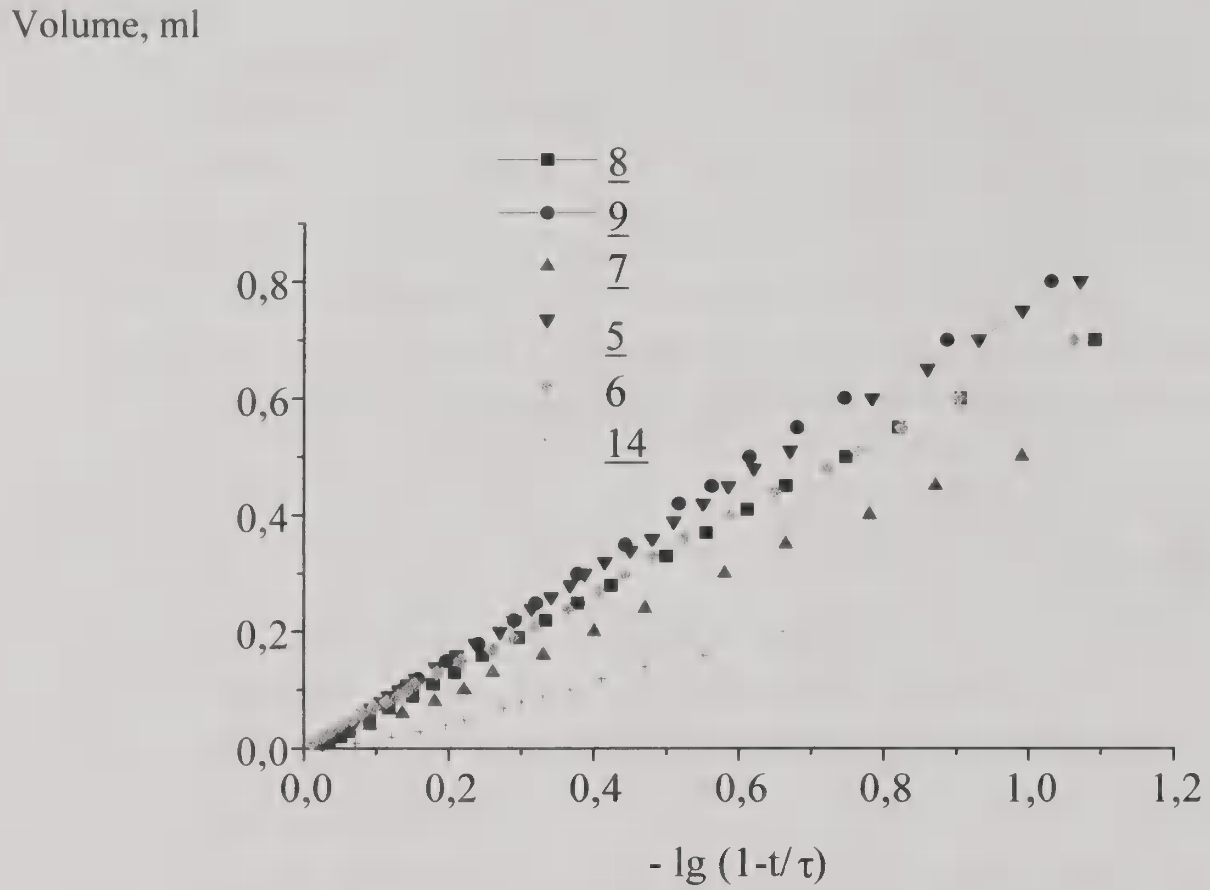


Figure 1. Half-logarithmic anamorphosis of compounds **5** - **9** and polyurethane **14**

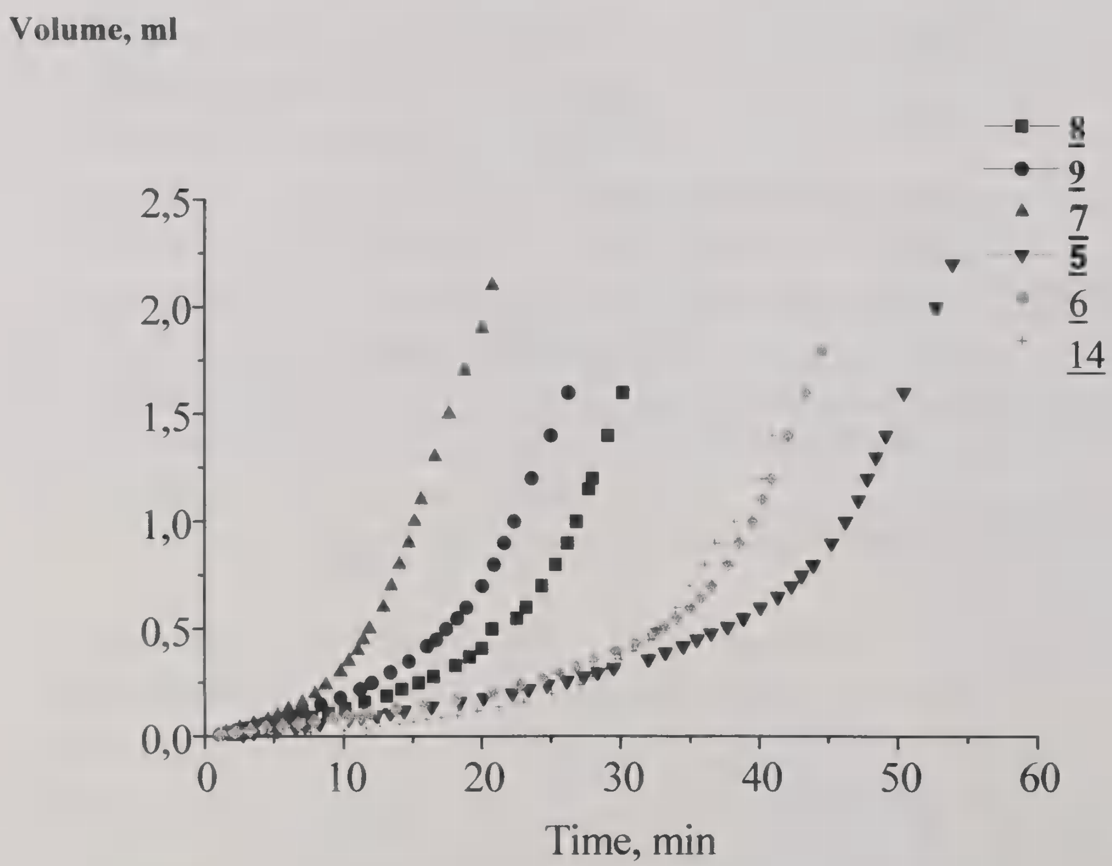


Figure 2. Kinetic curves of compounds **5**- **9** and polyurethane **14**

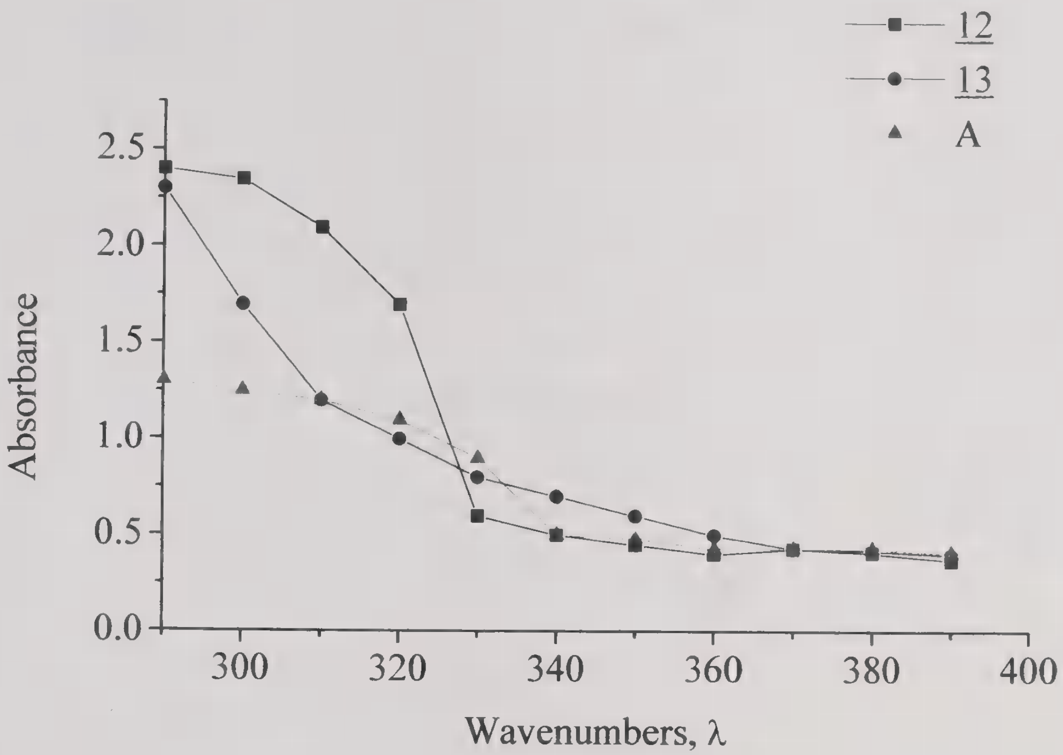
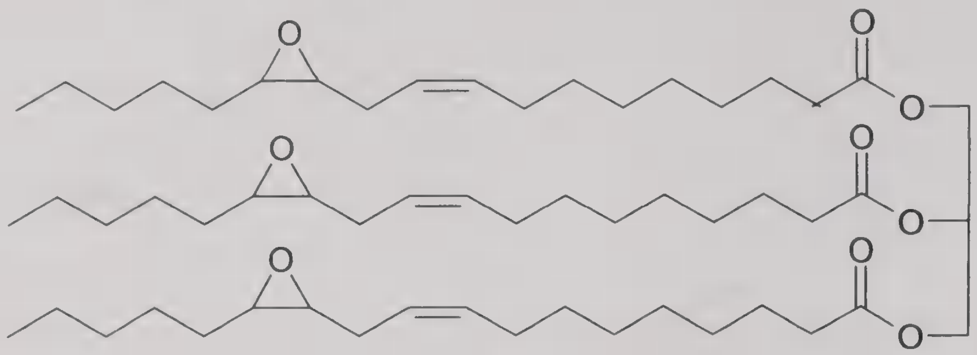


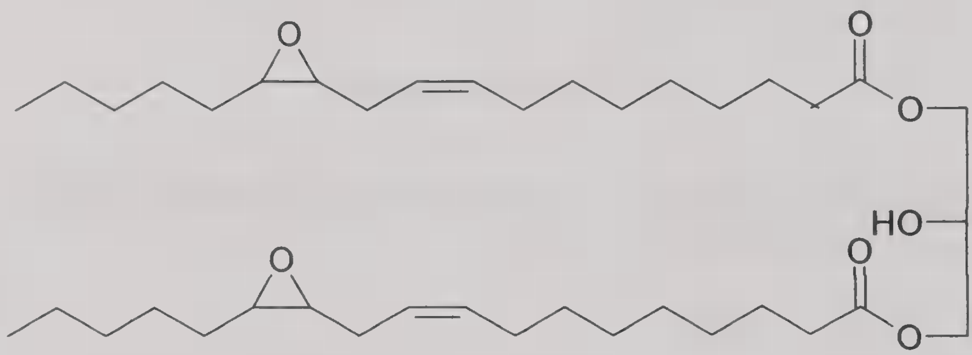
Figure 3 Comparison of commercial sunscreen product **A**, with the synthesized sunscreen products **12** and **13** ,

Figure 4 **Structures studied**



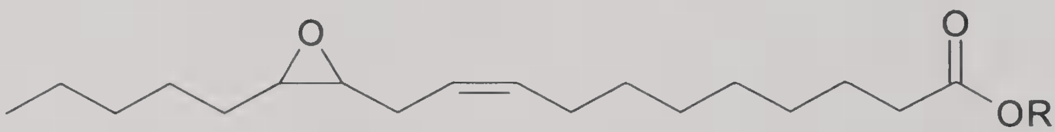
TRIVERNOLIN

1



DIVERNOLIN

4

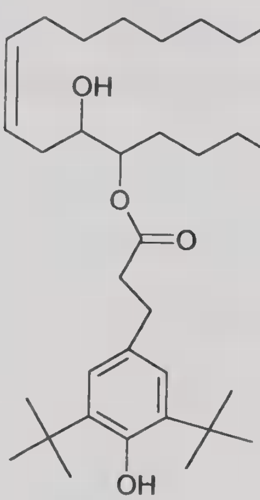


R	H	CH_3
	2	**3**

VERNOLIC ACID, 2; VERNOLIC ACID METHYL ESTER, 3

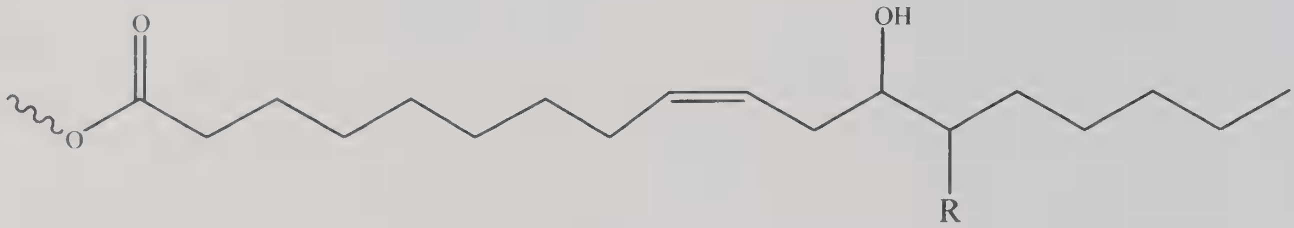
cis-13(12)-{3-(3,5-DI-*TERT*-BUTYL-4-HYDROXYPHENYL)PROPIONYLOXY}-12(13)-HYDROXYOLEIC ACID (**5**) AND

cis-METHYL13(12)-{3-(3,5-DI-*TERT*-BUTYL-4-HYDROXYPHENYL)PROPIONYL OXY} -12(13)-HYDROXYOLEATE (**6**)

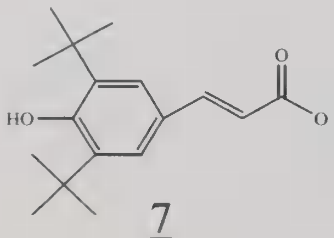


R	H	CH_3
	5	**6**

Figure 5 General Reaction Products



R = 7, 8, 9, 10, 11, 12 and 13



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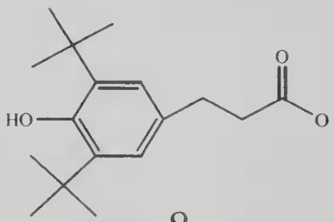

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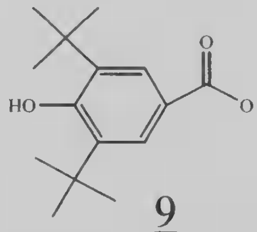

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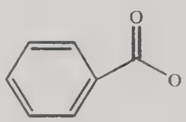

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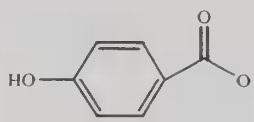

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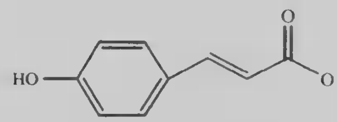

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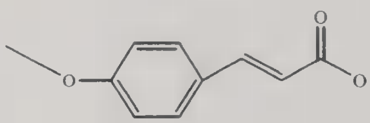

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Synthesis and Physical Properties of Some Symmetrical Disaturated Monounsaturated Triacylglycerols and Their Functional Roles in Food Oil Systems

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National Center for Agricultural Utilization Research
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50th Oilseed conference, New Orleans, LA, Jan. 21-23, 2000

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ABSTRACT

Symmetrical SUS triacylglycerols (TAGs), where S = saturated fatty acids (stearic (S)) and U = unsaturated fatty acids (oleic (O), linoleic (L), or linolenic (Ln)), were synthesized and their physical properties determined. The reaction sequence consisted of stearic acid transesterified to tristearin, followed by conversion of the TAG to a mixture of 1,3 and 1,2 diacylglycerols (DAGs) isolated and purified by crystallization and column chromatography. The pure 1,3 DAGs were then esterified with the appropriate fatty acid to form the symmetrical TAGs. SOS was 98.4% pure while the SLS and SLnS were 100% pure when analyzed by high pressure liquid chromatography (HPLC). The solid fat content (SFC) of the TAGs was determined by pulsed nuclear magnetic resonance spectroscopy over a temperature range of 10-50°C. Symmetrical disaturated TAGs are high, yet sharp melting compounds. For example, SOS, SLS and SLnS show high solids at temperatures of 30-35°C, yet are completely melted at temperatures only a few degrees higher. The drop melting points for SOS, SLS and SLnS were 44.1, 37.9 and 36.5°C respectively. Studies made on admixtures of the symmetrical SUS TAGs with soybean oil show typical linear SFC profiles, much like those observed with hydrogenated basestock/liquid oil blends used for margarine/spread formulations. Symmetrical disaturated TAGs are important components providing functionality to interesterified fat blends and structurally modified oils obtained by plant breeding since their high melting properties provide needed functionality at 10 and 21.1 °C (spreadability, resistance to water/oil loss at room temperature) while their sharply melting characteristics provide mouth feel and quick flavor release at body temperature. However, high stearic acid soybean oil contains insufficient amounts of SUS TAGs to furnish enough solid fat beyond temperatures of 10°C and requires conversion to less sharply melting SSU TAGs through interesterification of the glyceride structure.

INTRODUCTION

The functional properties of margarines/spreads including spreadability, resistance to water/oil loss, and melting at body temperature are, to a large extent, governed by the amounts and types of TAGs in the oil phase (1). Traditionally, functionality has been achieved through hydrogenation, interesterification, fractionation or blending of tropical oils and, more recently, through structural modification by plant breeding (2). Hydrogenation remains the technology of choice because of the sharp melting properties of the *trans* acids formed during the process. For

example, soybean oil, hydrogenated to iodine values of about 70 under selective conditions, yields a product having high solids at temperatures of 10-20°C, yet one which melts very sharply at body temperature. Such a basestock, when blended with liquid oil, yields oil suitable for incorporation into a wide variety of formulations including soft tub, spreadable stick and stick products.

Random interesterification of liquid oils consisting largely of triunsaturated and diunsaturated TAGs with trisaturated TAGs results in products consisting largely of mono and disaturated TAGs which provide the required functionality, i.e. melting point, solid fat content (SFC). Generally speaking, interesterification results in high, flat solid fat index/content profiles more suitable for producing shortening fats than margarine/spread oils where high, steep, sharply melting solid fat index profiles are desirable.

During the past decade, a number of structurally modified soybean oils, high in saturated acids, have been introduced and, after interesterification of their glyceride structure have been utilized in soft margarines (3-6). In their natural states, soybean oils, high in saturated acids, exhibit melting points about 20°C lower and lack sufficient solids beyond 10°C than the hydrogenated oils used in soft margarines. After random interesterification, melting points and solid fat content increase to values required for margarines. Since saturated fatty acids are essentially absent from the 2- position of structurally modified, but not interesterified, TAGs, the disaturated TAGs consist of the symmetrical SUS types (7,8). After interesterification, a portion of the SUS TAGs are converted to SSU types, which we suggested could account for the increased melting point and solid fat index profiles. This work was undertaken in order to characterize the physical properties of some symmetrical SUS disaturated TAGs and study their functional properties in food oil systems.

EXPERIMENTAL

Stearic (S), oleic (O), linoleic (L) and linolenic (Ln) acids were purchased from Nu-Chek-Prep (Elysian, MN) and were found 100% pure after conversion to methyl esters and analysis by gas-liquid chromatography. The high stearic acid soybean oils and their sources have been described previously (6,8). The conventional soybean oil was a refined, bleached, deodorized salad oil obtained from C&T Refinery (Charlotte, NC). The cocoa butter was supplied by K. Ritter (M&M Chocolate Co.). The symmetrical TAGs SOS, SLS and SLnS were synthesized from 1,3 distearyl glycerol according to method by Kodali, et al. (9)

<u>Preparation of tristearin.</u> Stearic acid (62.4 g, 0.22 moles) was combined with glycerol (6.08 g, 0.66 moles) and 1.4 g p-toluene sulfonic acid in a 200 ml, 3 necked flask fitted with an argon inlet and thermometer. The contents were stirred magnetically and heated by oil bath to 115°C. Water which condensed on the flask walls during the reaction was removed by use of a heat gun. During the course of the reaction, samples were taken and checked for conversion to the TAG by thin layer chromatography on 1 X 3" silica plates (Whatman) developed in benzene:diethyl ether (8:2) and visualized by 10%$CuSO_4$ in 8% H_3PO_4 spray and charring at 150°C on a hot plate. After 6 hours the mixture was filtered and the precipitate was discarded. The filtrate, containing tristearin, was crystallized in toluene at 4°C for 1.5 hours and then refiltered at 4°C. The crude tristearin was transferred to a flask with acetone, melted, and then allowed to cool slowly for 1 hour at room temperature. The slurry was then filtered at -20°C. The final product was dried

under vacuum at 40°C for 2 hours. The yield of tristearin was 55 g: 88% with a melting point of 73.5°C. The reported melting point is 73.1°C (10).

Preparation of 1,2 and 1,3 distearin. Tristearin (53.4 g, 0.6 moles) and glycerol (2.76 g, 0.03 moles) were combined as described above and 0.33 g sodium methoxide was added. The mixture was heated with stirring until thin layer chromatography showed that the DAGs were the predominant species. The DAGs were separated from the reaction mixture by column chromatography as follows. A large glass column packed with 400 g Baker 60/200 mesh silica gel was used. Residual TAGs were eluted with benzene, the 1,3 and 1,2 DAGs with ethyl ether/benzene (1:9) and MAGs with 100% ethyl ether. 1,3 DAG mixture was isolated from 1,2 DAG and any residual TAG, MAG and free fatty acids (FFA) by multiple crystallizations from acetone, diethyl ether and/or petroleum ether. The purified 1,3 DAGs were used to prepare the symmetrical TAGs as follows.

Preparation of SOS. 1,3 Distearyl glycerol (6.26 g, 0.01 moles) was dissolved in 150 ml carbon tetrachloride and placed in a 3 necked flask as described previously. Oleic acid (3.1 g, 10% excess) was added in 10 ml of carbon tetrachloride. 4-dimethyl amino pyridine (1.22 g) was added to the reaction vessel followed by the dropwise addition of 1.34 g N,N'-dicyclohexyl carbodiimide over a 30 minute period. The reaction was allowed to proceed at 28°C for 2 hours during which time a precipitate had formed. The precipitate was removed by filtration and the solvents, containing SOS, taken to dryness on a rotary evaporator. The crude TAG residue was purified by elution through a 1" diameter glass column packed with 70g silica gel. TAG were eluted with 5% diethyl ether in petroleum ether while DAGs, MAGs and residual FFAs were eluted with 100% diethyl ether. SLS and SLnS were prepared in a similar fashion.

HPLC analysis of symmetrical TAG . 50mg of the purified TAGs were dissolved in 2 ml methylene chloride and injected onto 2 reversed phase columns (Keystone, inertsil ODS2, 250x4.6 cm) connected in series. A Spectra Physics SP800 pump supplied a linear gradient of 80/20 acetonitrile/methylene chloride to 100% methylene chloride at 0.8 ml/min over 100 minutes. A Sedex 55 electron light scattering detector was used with data integration by Varian Star software. SOS was determined to be 98.4% pure (1.6% SSS) by HPLC: SLS and SLnS showed no impurities.

Nuclear magnetic resonance/SFC and melting point determination. Solid fat content (SFC) was determined by pulsed nuclear magnetic resonance spectroscopy according to official AOCS methods (11). The temperature range studied was from 10-50°C. A Bruker Minispec instrument was used. Drop melting points were determined according to AOCS methods (11) and values reported are the means of duplicate determinations.

Lipolysis. The method of Awl (12) was used with modification. TAGs (30 mg) were placed in a 1.6 X 12 cm capped tube along with 2 ml of 1M Tris buffer, 120 ul of 22% calcium chloride, 70 ul of 0.1% sodium cholate solution and 30 mg porcine lipase. The solution was stirred by vortexing for 20 seconds and the samples were incubated, with shaking, in a 37°C water bath. After 15 minutes, 10 ml of diethyl ether was added to stop the reaction. After vortexing the samples were allowed to stand 15 minutes. The organic layer (TAGs, DAGs, MAGs and FFAs)

was transferred to capped test tubes and evaporated to dryness under a stream of nitrogen. Residual water was removed by addition of acetonitrile and the solvent was evaporated under nitrogen. The lipolysate was placed on a micro silica column (Varian Bond Elut, 3cc, #1216-2037) that had been pre-rinsed with N-hexane. Ethyl ether (20 ml) was used to elute TAG and FFAs. DAGs were eluted with 3 ml ether:hexane:acetic acid (50:50:1). MAGs were eluted with methanol (4ml).

Preparation and analysis of methyl esters. The TAGs and MAGs formed by the lipolysis above were converted to fatty acid methyl esters and analyzed by gas-liquid chromatography as described previously (13).

Glyceride structure determination. TAG structures were determined from the lipase hydrolysis data and the 1,3 random-2 random theory advanced by Vanderwaal and Coleman (14, 15) where fatty acid composition of all possible chemically distinguishable isomers are predicted from fatty acid composition of the TAGs and the MAGs at the 2 position.

RESULTS AND DISCUSSION

The physical properties of the symmetrical TAGs synthesized in this study are shown in Table 1. The solid fat content, as determined by pulsed NMR spectroscopy over a 10-50°C temperature range, shows the interesting functional properties of these TAGs. At temperatures normally used to define the solid fat content of an edible fat or oil, SOS, SLS and SLnS exhibit high and sharp melting points. SOS, SLS and SLnS all show high amounts of solid fat at temperatures up to 30°C, yet are completely melted at 36-44°C. Dropping points for SOS, SLS and SLnS were 44.1, 37.9 and 36.5°C respectively. The data clearly demonstrate the effect of unsaturation on melting points of the SUS TAGs. Tristearin melts at 73.5°C, where one double bond/TAG (SOS) results in a decrease of 29°C, two double bonds 35.6°C (SLS) and three double bonds (SLnS) 37°C. The melting point of SOS has been reported as 41.6 (16), 42.5-43 (17) and 43.5°C (18) respectively. The melting points of SLS and SLnS have not been reported in the literature.

TABLE 1
Physical properties of structured triglycerides.

Triglyceride	Purity (%)	Solid Fat Content @ Temp. (°C)[a] 10	21.1	26.7	33.3	40	45	50	Drop Melting Point (°C)[b]
SSS	100	96.6	96.6	96.6	96.5	96.5	96.6	96.7	73.5
SOS	98.4	93.1	91.8	91.2	96.7	86.0	3.8	1.3	44.1
SLS	100	88.5	87.8	87.5	78.7	0.0	0.5	0.1	37.9
SLnS	100	97.2	96.6	90.9	73.5	0.2	0.2	0.0	36.5

[a]By pulsed NMR, AOCS method
[b]AOCS method

The symmetrical SUS TAG content of a number of high stearic acid soybean oils are shown in Table 2. Total SUS TAGs range from about 14-30% with the major components consisting of SOS (1-5%), SLS (4-13%), SOP (1-3%) and SLP (4-6%). Since the linolenic acid content of high saturate soybean oils is low, SLnS is a minor TAG seldomly exceeding 1%.

In order to more fully characterize the functional properties of the TAGs synthesized in this study, blends of SOS, SLS and SlnS and ordinary soybean oil were prepared and their solid fat content and drop melting points were determined. Results are shown in Table 3. The results indicate that high stearic acid oils should contain 10-25% of SOS or SLS in order to possess enough solid fat for margarines/spreads and have a drop melting point in the desired 31-32°C range. However, only the A-6 varieties developed at Iowa State University approach these approximate values.

TABLE 2
Symmetrical SUS triglyceride content of high stearic acid soybean oils.[a]

Sample	% Stearic Acid	SUS Triglycerides POP[b]	PLP	PLnP	SOS	SLS	SLnS	SOP	SLP	SLnP	Total SUS Triglycerides (%)	Drop Point (°C)
1	17.2	0.2	1.0	0.2	1.0	4.7	0.7	1.0	4.3	0.7	13.8	14.2
2	16.3	0.7	1.5	0.1	1.7	3.9	0.3	2.1	4.8	0.4	15.5	14.6
3	18.3	0.5	1.3	0.1	2.0	5.2	0.4	1.9	5.1	0.4	16.9	16.3
4	20.6	0.4	0.9	0.1	2.5	6.5	0.5	1.9	4.9	0.4	18.0	17.6
5	23.9	0.4	0.9	0.1	2.9	7.7	0.9	2.0	5.4	0.7	21.0	16.4
6	28.2	0.3	0.9	0.1	4.2	11.1	1.3	2.4	6.3	0.7	27.4	14.2
7	30.3	0.3	0.8	0.1	4.9	12.7	1.4	2.4	6.4	0.7	29.7	18.7

[a]By lipase hydrolysis and 1,3-random, 2-random theory.
[b]Palmitic

TABLE 3
Solid fat content of blends of structured triglycerides and soybean oil.

Triglyceride	% Mixed With Soybean Oil	Solid Fat Content @ Temp. (°C)[a] 10	21.1	26.7	33.3	40	45	50	Drop Melting Point (°C)[b]
SOS	0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	-14.3
	5	5.2	2.6	0.9	0.2	0.5	0.0	0.0	10.6
	10	8.5	5.3	3.3	0.7	0.0	0.0	0.0	15.0
	15	13.3	9.9	6.3	1.3	0.2	0.0	0.0	26.5
	20	12.4	10.5	7.5	2.1	0.2	0.0	0.0	29.0
	25	14.3	13.0	9.9	3.4	0.6	0.0	0.0	30.7
	100	93.1	91.8	91.2	96.7	86.0	3.8	1.3	44.3
SLS	0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	-14.3
	5	4.4	0.0	0.0	0.0	0.0	0.0	0.0	16.6
	10	9.6	0.6	0.0	0.0	0.0	0.0	0.0	20.2
	15	14.1	5.8	0.0	0.0	0.0	0.0	0.0	22.8
	20	17.6	10.2	0.0	0.0	0.0	0.0	0.0	24.1
	25	24.7	17.4	5.1	0.3	0.0	0.0	0.0	26.6
	100	88.5	87.8	87.5	78.7	0.0	0.5	0.1	37.9
SLnS	0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	-14.3
	5	4.9	0.2	0.2	0.0	0.0	0.0	0.2	18.2
	10	8.4	4.7	0.0	0.0	0.3	0.0	0.4	19.8
	15	17.5	12.7	6.6	0.5	0.2	0.0	0.7	22.8
	20	22.2	18.8	12.5	0.1	0.0	0.4	0.0	24.9
	25	25.8	23.2	16.8	1.9	0.2	0.3	0.0	26.2
	100	97.2	96.6	90.9	73.5	0.2	0.2	0.0	36.5

[a]By pulsed NMR, AOCS method
[b]AOCS method

Cocoa butter is, perhaps, the best example where TAG structure can be related to functional properties. Cocoa butter consists of 3 TAGs comprising nearly 80% of total TAG. These TAGs include the symmetrical SUS TAGs POP (P=palmitic), POS and SOS in the proportions 16%, 37% and 25% respectively. Cocoa butter shows the following SFC profile: 10°C 88.4, 21.1°C 79.2, 26.7°C 69.4, 33.3°C 10, 40°C 0.4 and has a melting point of 29°C. At room temperature cocoa butter is a solid material, yet it melts very sharply at body temperature. The latter characteristic can be attributed to the high concentration of symmetrical TAGs with melting points between 35-44°C. POP and SOP would be expected to show SFC profiles similar to those shown for SOS, SLS and SLnS (Table 1).

The solid fat content and drop melting points of blends of SOS and liquid soybean oil are shown in Figure 1 and illustrate the impact of the low melting TAGs contributed by the liquid soybean oil. Pure SOS shows the high, but sharp, melting properties previously discussed, while the blends assume more of a linear relationship between solid content and temperature (part A). Part B, which shows the effect of SOS on drop melting point, illustrate that blending a structured, high, but sharply melting, TAG with low melting TAGs produces a dramatic increase in melting point. Pure soybean oil shows a drop melting point of -14.3°C. At concentrations of 25% SOS, the drop melting point has increased to about 31°C, but the addition of 75% additional SOS results in only a 13°C rise in melting point. Similar results (data not shown) were observed with blends of cocoa butter and soybean oil TAGs. These results, in part, explain why structurally modified, high stearic acid soybean oils do not exhibit the solid fat content and melting points required for margarine/spread formulation. The low concentration of sharply melting SUS TAGs in these oils (Table 2), coupled with the dilution effect from low melting TAGs are evident from Figure 1. We have postulated that increases in solid fat and melting points after

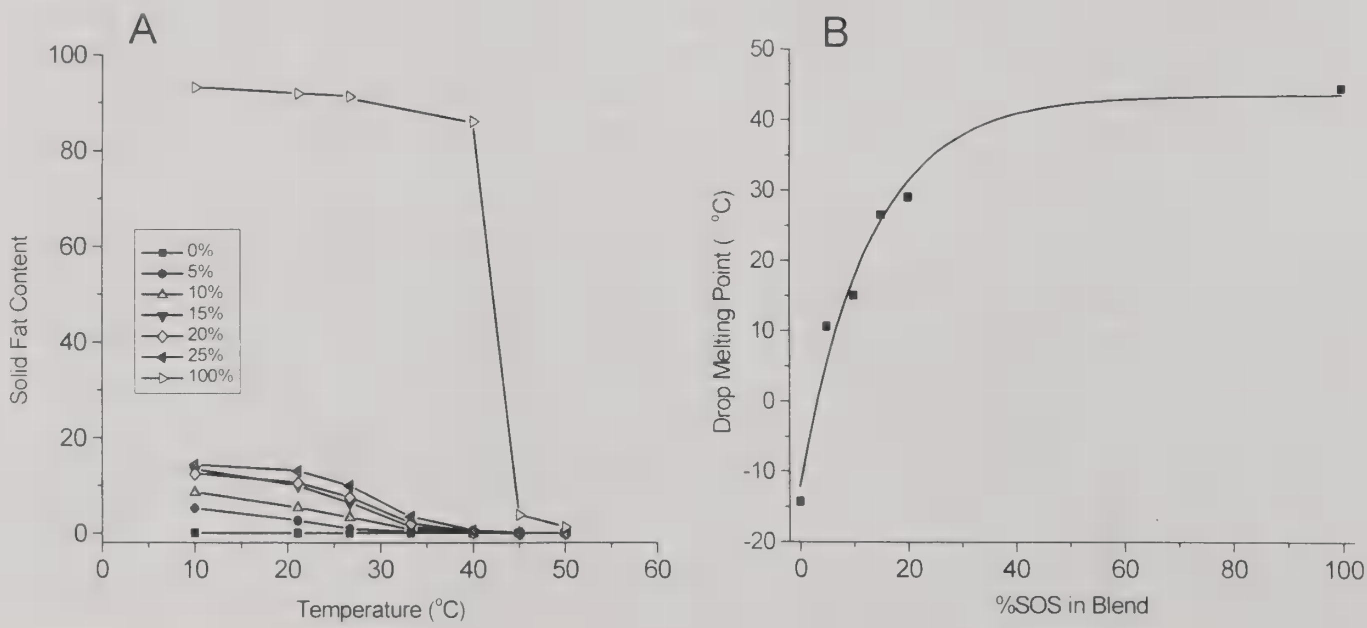

Figure 1. Effect of blending SOS and soybean oil on solid fat content and drop melting points.

interesterification of high stearic acid oils results from conversion of symmetrical SUS TAGs to the non-symmetrical SSU types. (6) Synthesis and further characterization of SSO. SSL and SSLn is underway and should provide additional information on the behavior of functional TAGs in food oil systems.

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A Comparison of Corn Oils: Corn Germ Oil, Corn Kernel Oil, and Corn Fiber Oil

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A Comparison of Corn Oils: Corn Germ Oil, Corn Kernel Oil, and Corn Fiber Oil

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Commercial corn oil is obtained by extracting corn germ and it could more accurately be described as "corn germ oil". Corn germ is a high-valued "co-product" obtained during the wet milling of corn, an industrial process designed to economically fractionate corn kernels into starch, oil, protein, and fiber fractions. Corn fiber is a low-valued co-product of wet milling and it is currently mixed with other components and sold as "corn gluten feed", an inexpensive ($ 0.03-0.05 /lb) animal feed. Recently, we have reported a new type of healthy corn oil, corn fiber oil, that can be extracted from corn fiber. Corn fiber oil contains the highest levels of phytosterols and phytostanols (saturated phytosterols) of any known oil obtained from natural sources, levels that are similar to those in the phytosterol-enriched margarine products that are currently being marketed in the US. Although corn kernels contain relatively low levels of oil (3-4%), and are not considered oilseeds, an oil, "corn kernel oil", can be obtained by directly extracting corn kernels and this oil has intermediate levels of phytosterols, and its commercial value should be considered. The levels of total phytosterols in corn germ oil, corn kernel oil, and corn fiber oil, are approximately 1, 2, and 12%, respectively.

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Expression of Three Omega-3 Fatty Acid Desaturase Genes from Tung Seeds

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Abstract
Polyunsaturated fats are important for plant responses to temperature changes and pests, as well as in human nutrition. Low levels of polyunsaturated fats in the human diet have been shown to lead to cardiovascular problems. We have found genes which may be used to convert plant oils into oils with new properties by increasing the amount or changing the type of polyunsaturated fats in the oils. Three genes related to omega-3 fatty acid desaturase, an enzyme which adds a double bond (unsaturation) to fatty acids, have been identified in cDNA clones made from tung seed RNA. We have analyzed the expression pattern using semi-quantitative RT-PCR. All three genes are expressed in both seeds and leaves. However, one is expressed more strongly in seeds, one more strongly in leaves, and one is expressed at a low level in both seeds and leaves. The use of the genes reported here in microorganisms may lead to new or improved oilseed products.

Introduction
Omega-3 fatty acid desaturase enzymes change the polyunsaturation level of fatty acids from two double bonds (such as linoleic acid) to three double bonds (such as linolenic acid). Polyunsaturated fatty acids are found in plant oils and membranes and now thought to be an essential part of the plant response to wounding (Kodama et al, 2000; Martin et al, 1999; Nishiuchi et al, 1999) and temperature changes (Gibson et al, 1994; Kanervo et al, 1997; Murakami et al, 2000). In humans, polyunsaturated fatty acids are essential in the diet (Hornstra, 2000) and presence of insufficient quantities in the diet is linked to cardiovascular symptoms (Charnock, 1999; Veicel et al, 1999). In plants, production of polyunsaturated fatty acids has been found in both endoplasmic reticulum and in chloroplast membranes, and a number of genes have been cloned (Yadav et al, 1993).

We have cloned three different omega-3 type fatty acid desaturases from tung seeds, TnDES-1, -2, and -3. TnDES-1 has characteristics of endoplasmic reticulum-localized proteins (Benghezal et al, 2000) and is 387 amino acids in length. The remaining two are both close to 450 amino acids in length and show strong homology to chloroplast proteins. TnDES-2 has a chloroplast transit sequence, that portion of a protein which directs it to the chloroplast. The amino acid sequence comparison among the three genes reveals 55% to 65 %identity, with very strong similarity in sequence areas common to all omega-3 desaturases (Shepherd et al, 2000). All three

show approximately 70% amino acid sequence identity with a plastid-type gene isolated from cDNA to castor bean seed (van de Loo and Somerville, 1994). To further clarify how the three tung seed genes differ and to determine their function in the growth and development of the plant, we have analyzed the expression pattern of the three genes during seed maturation and in leaves.

MATERIALS AND METHODS

Tung fruits were collected from the American Tung Oil Corporation orchard in Lumberton, MS during the early morning hours on several dates between the time of blooming and the time of fruit maturity. Seeds were excised in the field and immediately frozen in liquid nitrogen, then stored at -80 C. Young, fully expanded leaves were collected and frozen at the same time.

For RNA isolation, tung nuts or leaves were ground in liquid nitrogen, then RNA was extracted using the hot borate method of Wan and Wilkins (1994). RNA was stored in water at -80 C. Primers were designed based on dissimilar portions of the sequences of the three desaturase genes. The primers were:
TnDES1: 5'-TAAGCAATCCTCCTCCAT-3' and 5'-CAACTTGCTTTTATCAAC-3'
TnDES2: 5'-TTCCAAAATCTACTCTA-3' and 5'-TGAAGAGGCATCGAGCTCC-3'
TnDES3: 5'-ATCTAAAACTCTCTTCTA-3' and 5'-TATTAACCTATGCAAAT-3'

RT-PCR was performed using a dilution series of RNAs to determine the comparative level of transcript which was present for the three genes in seeds of different ages and in leaves. Reactions were carried using 2 ug, 0.2 ug, 0.02 ug, and 0.002 ug of total RNA using the Advantage One-Step RT-PCR kit (Clontech, Palo Alto, CA). Temperature conditions used for the reactions were 50 C for 1 hr for the reverse transcriptase reaction, then 94 C for 5 min followed by 30 cycles of: 94 C for 30 sec, 55 C for 30 sec, 68 C for 90 sec. Products of the reactions were analyzed on 1% agarose gels in TBE buffer (Sambrook, et al, 1993).

RESULTS

Using primers which were based on unique sequences in each of the three genes, the level of expression of each gene during the maturation time of tung seeds was analyzed, as well as expression in leaves. Results for the genes and time points are summarized in Table 1. Time points are the dates of seed or leaf collection. Fatty acid production in seeds begins in early August and continues through September. Results are shown as the number of dilutions at which a product was made by the reaction for each collection date. Thus, those with one "+" were detected only in the undiluted RNA sample, "++" in the 1/10 dilution, etc. This should be directly related to the abundance of mRNA for the particular gene which is present in the RNA sample, and thus should be directly related to abundance of the protein at the different developmental stages.

DISCUSSION

We have used a semi-quantitative RT-PCR method (Zheng et al, 2000) to compare the levels of mRNAs for three different fatty acid desaturases present in seeds and leaves of tung trees. All three genes are expressed throughout most of tung seed development and in leaves, but at different levels. None of the expression patterns correlate strongly with oil and fatty acid accumulation patterns in the seed. TnDES1, presumed to be localized in the endoplasmic reticulum based on targeting sequences seen in the protein sequence derived from the nucleotide sequence, is expressed highest in seeds and lower in leaves. TnDES2, presumed to be localized in the chloroplast based on the presence of a transit peptide sequence, has highest expression in leaves, but is also found in seeds. TnDES3 is found at low levels in both seeds and leaves.

Interestingly, all three genes share a 70% identity at the amino acid level to castor bean chloroplast omega-3 desaturase (van de Loo and Somerville, 1994), although one (TnDES1) shows no evidence of being plastidic. They are all more similar to the castor bean gene than they are to each other. This is no doubt due to the similarity of all omega-3 desaturases and in this case ones that were all derived from mRNA found in seeds.

The finding of mRNA for chloroplast proteins in the seed is not as strange as it at first seems if one remembers that the cotyledons in the seed become the first leaves of the new plant and the development of the structures may already be taking place. The presumed chloroplast protein genes are expressed later, in the more mature seed, closer to the time of germination. In addition, the chloroplast proteins in other plants have been found to be related to wounding (Martin et al, 1999; Kodama et al, 2000) and temperature responses (Gibson et al, 1994; Murakami et al, 2000) so they may be active at times other than just in normal leaf formation and functioning (Kanervo et al, 1997).

The results indicate that the genes probably serve different purposes in the plant, and although the function is not certain, more evidence is now available. We plan to continue with heterologous expression of the genes in yeast to determine the reaction which is directed and the potential for new products. Polyunsaturated fatty acids are important components of several aspects of human health and market demand is growing (Alonso and Maroto, 2000). The use of genetic engineering to increase the amounts or types of polyunsaturates is being considered. The use of the genes reported here in conjunction with heterologous hosts (Reed et al, 2000) may lead to new or improved oilseed products.

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Table **Detection of expression in seed and leaf RNAs**

	Collection Dates			
Genes	7/15 seed	8/6 seed	8/17 seed	7/15 leaf
TnDES1	+	++	+	+
TnDES2	+	+	++	+++
TnDES3	+	+	+	+

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Production of Monolaurin from Coconut C12 Fatty Acid

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Bench Scale Production of Monolaurin from Coconut C_{12} Fatty Acid

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ABSTRACT

A method was developed for the esterification of coconut C_{12} fatty acid with glycerol to produce monolaurin in good yield and quality. The reaction parameters, i.e. molar ratio of the reactants in 1:2.5 (F.A.:Gly), temperature of 180 °C, 5 hours reaction time were optimized to obtain a product in 55.2% yield with 98.50% purity. Three methods of purification, namely Water Refining, Low Temperature Crystallization and Molecular Distillation, were studied and developed in this process. Among the three, the latter gave the highest quality monolaurin. The degree of purification of the product has been found to be a limiting factor to conform with the standard specification and requirement such as its percentage monolaurin content and its melting point for its various applications such as emulsifier, shortenings, preservatives, cosmetics, ointments and medicine. Fatty acid composition of the raw materials and the monolaurin, dilaurin and trilaurin were determined by gas chromatography. The refined monolaurin and standard monolaurin were determined and characterized by gas chromatography, high pressure liquid chromatography, infra red spectroscopy and nuclear magnetic resonance for comparison.

INTRODUCTION

In 1968, Babayan introduced medium chain triglycerides (MCT) which is known for its efficient and quick source of energy. Structured fats were also introduced by Babayan (1985). This structured fats with a MCT backbone with built-in essential fatty acid i.e. linoleic acid is best for patients, particularly the critically ill. These MCT and structured fats, which were both prepared from the esterification of medium chain fatty acid (C_8-C_{10}), had shown and manifested the nutritional and therapeutic effects of coconut oil. However, after several decades of research it was demonstrated that lauric acid, C_{12} is one of the truly beneficial among the medium chain fatty acids. It is the fatty acid found in human milk that is known to have unique anti-microbial properties; its presence in milk keeps infants from being infected by many viruses, protozoa, and some bacteria (Hamosh 1991; Thormar 1987). Several decades of research have demonstrated that "Lauric acid is one of the best inactivating fatty acids and its monoglyceride form is even more effective than the fatty acid alone" (Enig 1994). The digestive tract converts lauric acid into the powerful anti-microbial monoglyceride called monolaurin. Monolaurin is formed in the

digestive tract only when lauric acid is available in the diet (Hamosh 1991; Thormar 1987). Monolaurin inactivates envelope viruses (Thormar 1987), such as the HIV virus, cytomegalovirus, herpes virus, sna virus, as well as numerous bacteria and protozoa (Hamosh 1991) that cause opportunistic infections in individuals who are immune-suppressed.

Being one of the largest region of coconut plantation and largest exporter in Asia, and coconut oil as having the largest constituent of lauric fatty acid (about 48%), which its potentials as anti-microbial, anti-viral, and anti-protozoal have not been yet explored, this study has developed and established a process for the production of a low volume but high value monolaurin from lauric acid of coconut oil. The optimum conditions for the esterification of lauric acid with glycerol were established to obtain purified monolaurin with 55.2% yield based on the theoretical yield and 98.50% purity. The physicochemical properties of this monolaurin were determined and evaluated in comparison with the standard specification. Purification studies were also conducted to obtain a product that will conform to the specific requirements of the pharmaceutical industries, particularly for medicinal and therapeutic applications.

MATERIAL AND METHODS

Lauric acid was provided by Cocochem (Philippines). Its typical analysis as starting raw material is shown in **Table 1**. Standard monolaurin with the commercial name of Monolauroyl-rac-Glycerol, 99% pure, is a product of Sigma and purchased from Daimar Enterprises, Inc. Glycerol and other analytical reagents, i.e acetone, acetonitrile and isopropyl alcohol, used in the analysis were also purchased from local chemical suppliers (Merck, Instruchem, Chemline, etc.).

Table 1. Typical analysis of coconut lauric acid used as starting material

Saponification value, mg	279
Acid value, mg	279
Iodine value, mg	0.02
Melting temperature, °C	43.5
Lovibond color (5 ¼ cell)	0.6 red 2.2 yellow
Moisture, %	0.06
GC analysis, %	0.8, 99.0, 0.2

EXPERIMENTAL PROCEDURE/METHODOLOGY

Coconut C_{12} fatty acid was esterified with glycerol using the JICA donated 50-liter capacity Glass Lined Stirred Reactor. The established conditions such as the molar ratio, reaction temperature and time were used in the process. Routes in the esterification of glycerol are shown in **Figure 1**, while the process flowchart is shown in **Figure 2**.

The crude product obtained from the esterification was subjected to purification processes such as the molecular distillation, low temperature crystallization, and water-refining. The comparison of percentage purities and melting points of the product is shown in **Table 2**.

Molecular Distillation – Crude monolaurin was subjected to molecular distillation method. The equipment used was Centrifugal Molecular Still, Model CEH-300B, Yamato. Three kilograms of crude monolaurin was charged to the equipment. The distillation temperature for the non-reacted components was 75 °C, while that for the monolaurin was 95 °C.

Low Temperature Crystallization – Crude monolaurin was dissolved using acetone as solvent. One hundred grams of crude monolaurin was mixed with 400 ml acetone. The solution was stirred moderately until it attained homogeniety. The homogenous solution was immersed in a cold bath of about 15 °C temperature while stirring until it crystallized. The crystallized product was filtered and air-dried for 1 to 2 days.

Water-Refining – Crude monolaurin was washed in a separatory funnel with lukewarm brine solution (5%). The brine was added to 500 ml lukewarm water and this solution was added to the crude monolaurin, the mixture shaken and settled for a few minutes or until two layers had completely separated from the lower layer. The lower layer was discarded and an anhydrous sulfate (5%) was added to the upper layer and mixed. The mixture was then settled for about an hour. The final product was filtered and air-dried for 1 to 2 days.

Esterification of Lauric Acid with Glycerol

Complete esterification was conducted in a three-necked glass reactor with a thermometer, substripped with nitrogen gas, and a water condenser to distill out the water by product. One mole of fatty acid was reacted with an excess of the equivalent mole of the glycerol at specified reaction temperature and time. The schematic diagram of the esterification process is shown in **Figure 2**.

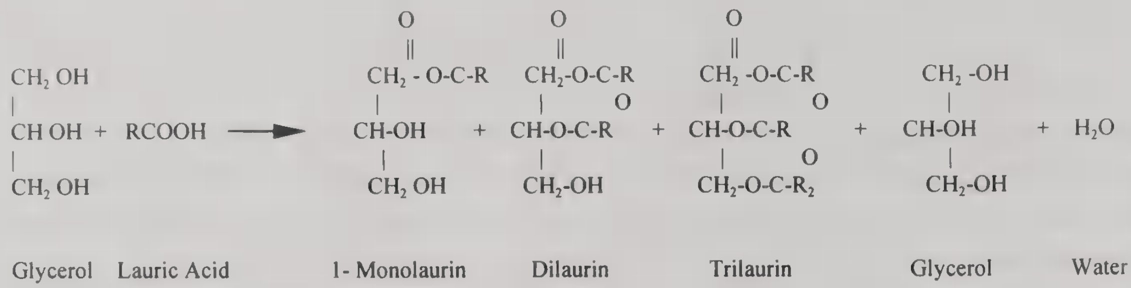


Figure 1. Routes in the esterification of glycerol.

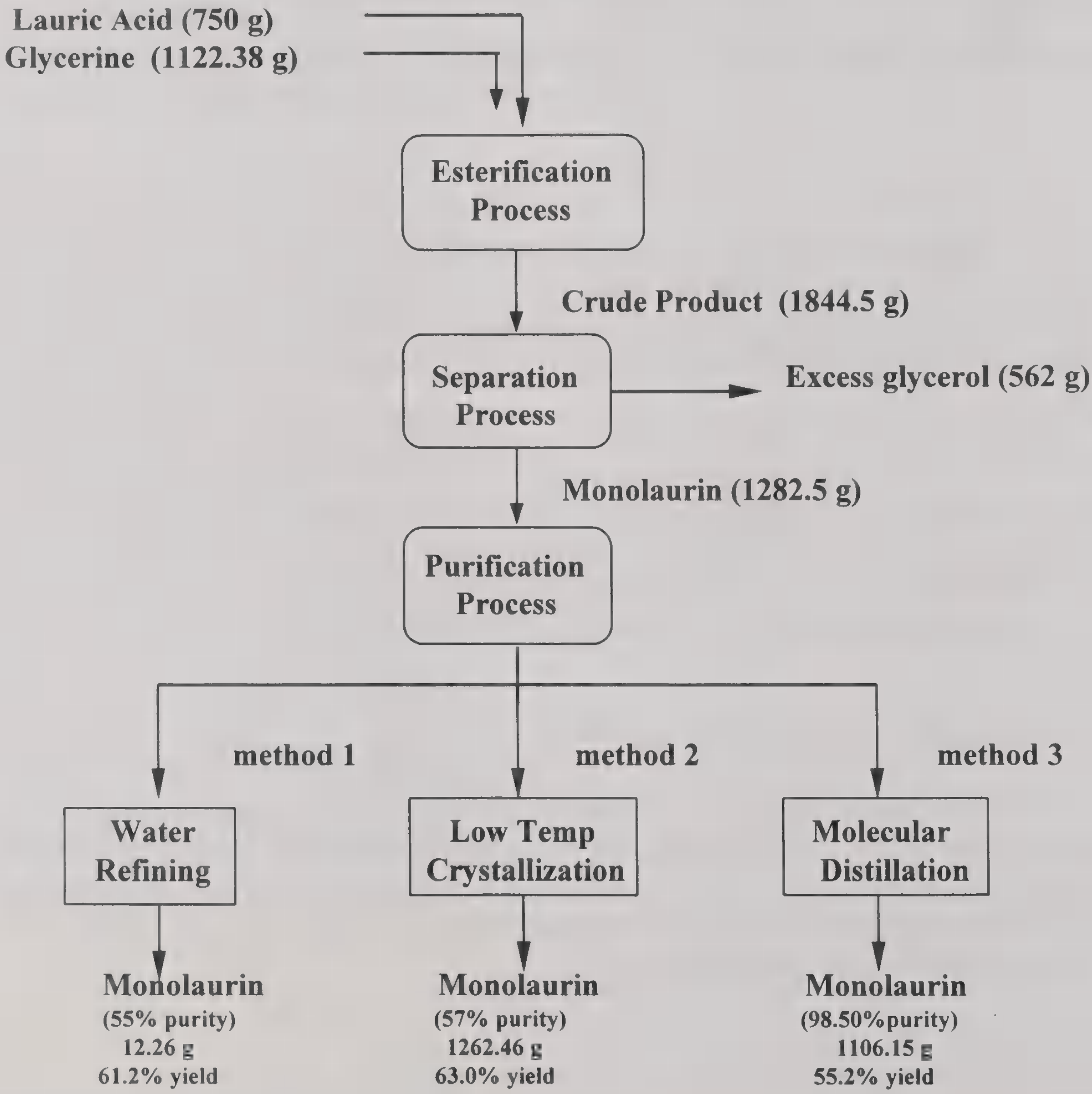


Figure 2. Scheme of typical monolaurin processing.

Table 2. Comparison of product obtained using different methods of purification.

Method of Purification	% purity	Melting Point, °C
Molecular Distillation Method	98.50	63.0°C
Low Temp Crystallization Method	57.00	43.0°C
Water Refining Method	55.00	43.0°C

Standard Monolaurin: Purity: 99%; Melting point: 63 °C

Analytical Methods for Monolaurin Characterization

Fourier Transform InfraRed Spectroscopy (FTIR) – The purified monolaurin was analyzed and characterized using Perkin Elmer FTIR Spectroscope, model 1720x, employing the diffused reflectance technique. FTIR spectra of both the standard and ITDI monolaurin are shown in **Figure 3**. The FTIR spectrum of the Sigma monolaurin standard is almost identical with the ITDI monolaurin. Esterification of lauric acid into monolaurin is evident by the appearance of a sharp band at 1186 cm^{-1} and a weak band at 1051 cm^{-1} for the C–O–C of the monolaurin formed.

Gas Chromatograph (GC) – The compositions of the product obtained from the esterification and the unreacted fatty acid were determined using a Shimadzu GC-14 gas chromatograph. One microliter volumes of the sample and the standard were injected into a OV-1 capillary column at a programmed temperature of 180 °C to 230 °C. Through flame ionization detection (FID), the components of the sample mixture and of the standard were detected with high purity nitrogen as the carrier gas. Comparative GC chromatogram of both the standard and ITDI monolaurin are shown in **Figure 4**.

High Pressure Liquid Chromatograph (HPLC) – A 3.0% purified monolaurin in eluent solution composed of heptane/isopropyl alcohol/acetonitrile, (88:10:2) was subjected to HPLC analysis using a Shimadzu HPLC, model LC-6A, to determine its percentage purity. It was characterized and compared with Sigma standard monolaurin, 99% purity. A 10 μL sample was injected into a Lichrocart 250-4 column using UV-VIS spectrophotometer detector at 210 nm @ 0.04x. Comparative HPLC chromatograms of both the standard and ITDI monolaurin are shown at **Figure 5**.

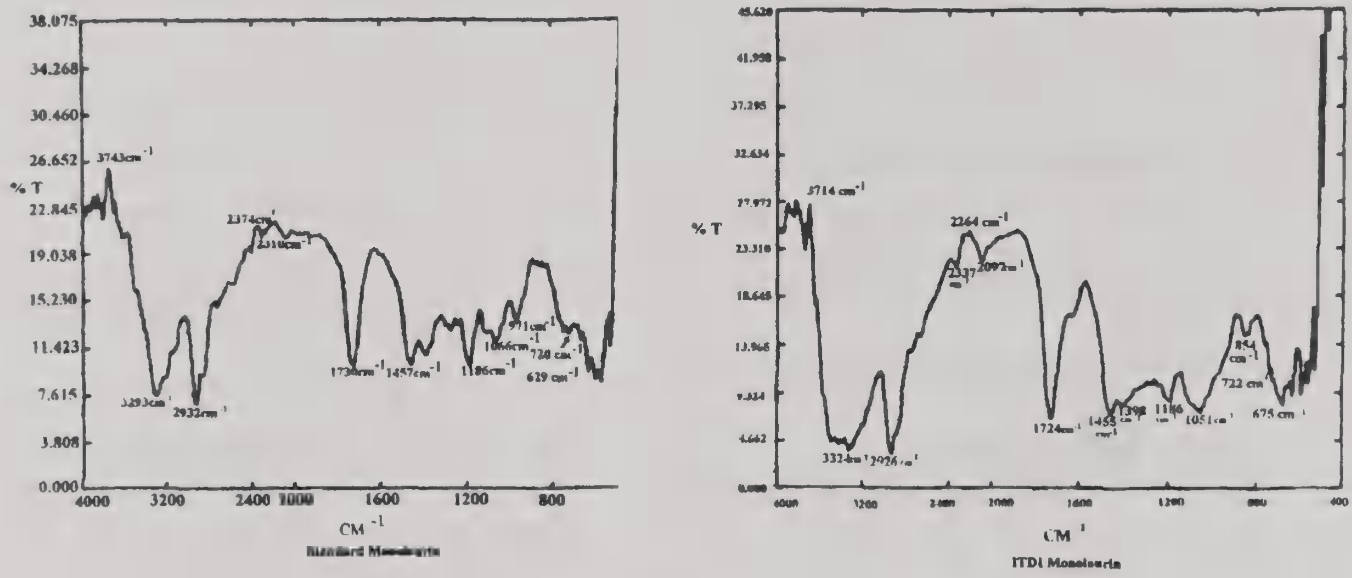


Figure 3. Comparative spectra of standard monolaurin and ITDI monolaurin.

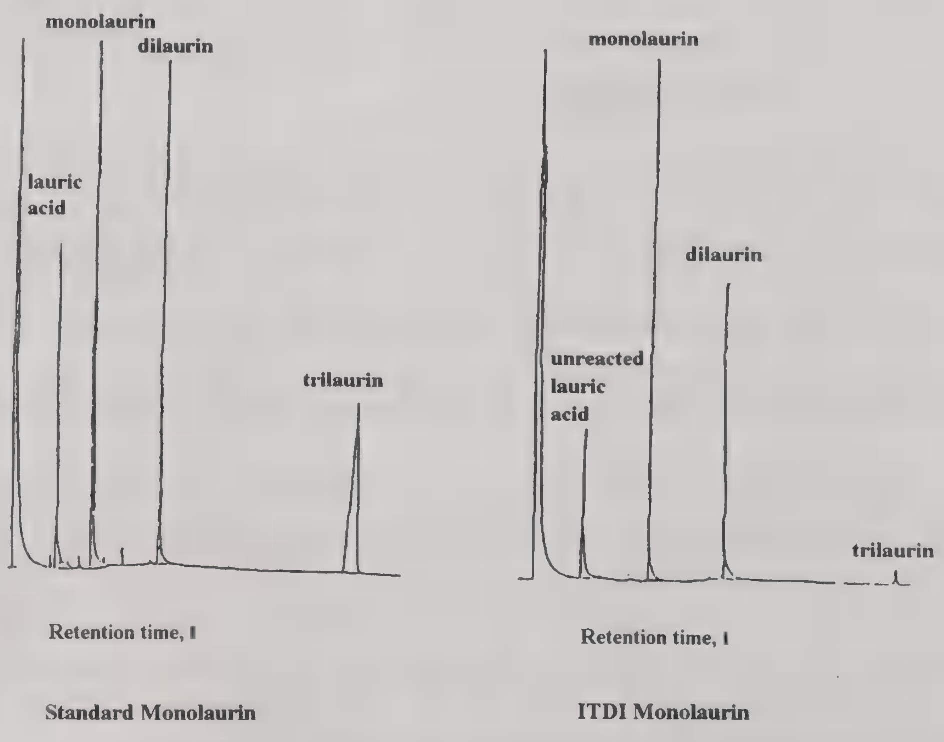


Figure 4. Comparative GC chromatogram of standard mixture and ITDI crude monolaurin.

Nuclear Magnetic Resonance (NMR) – The NMR analyses were performed on a JEOL Lambda 400 NMR spectrometer, observing ^{1}H at 399.65 MHz and ^{13}C at 100.40 MHz. NMR probe used was a 5 mm tunable probe (TH5). The solvent was benzene-D_6, and chemical shifts, δ, were referenced to tetramethyl silane (TMS).

The following NMR experiments were performed: ^{1}H, ^{13}C, and 2-dimensional ^{13}C-^{1}H heteronuclear shift correlation. The ^{1}H and ^{13}C assignments are summarized in **Table 3** and presented in **Figure 6**. These shift assignments are supported by the heteronuclear correlation experiment.

Table 3. The ^{1}H and ^{13}C assignments from NMR Analysis of monolaurin.

^{1}H assignments:

Chemical Shift,δ	**Multiplicity**	**Integration**	**Assignment**
4.56	broad	2	-OH
4.27	multiplet	2	diastereotopic protons on C1 of glyceryl group
4.01	broad multiplet	1	proton on C2 of glyceryl group
3.69	doublet of multiplets	2	diastereotopic protons on C3 of glyceryl group
2.27	triplet	2	methylene protons on C2 of lauryl group
1.63	broad multiplet	2	methylene groups on C3 of lauryl group
1.29- 1.26		16	methylene groups on C4-C11
0.91	triplet	3	terminal methyl on lauryl group

^{13}C assignments:

Chemical Shift,δ	**Assignment**	**Chemical Shift,δ**	**Assignment**
174.1	-O-CO- ester carbon	34.3	C2 of lauryl group
70.7	C2 of glyceryl group	34.3-29.6; 23.1	methylene carbons on lauryl group
65.4	C1 of glyceryl group	25.3	C2 methylene on lauryl group
63.8	C3 of glyceryl group	14.3	terminal methyl on lauryl group

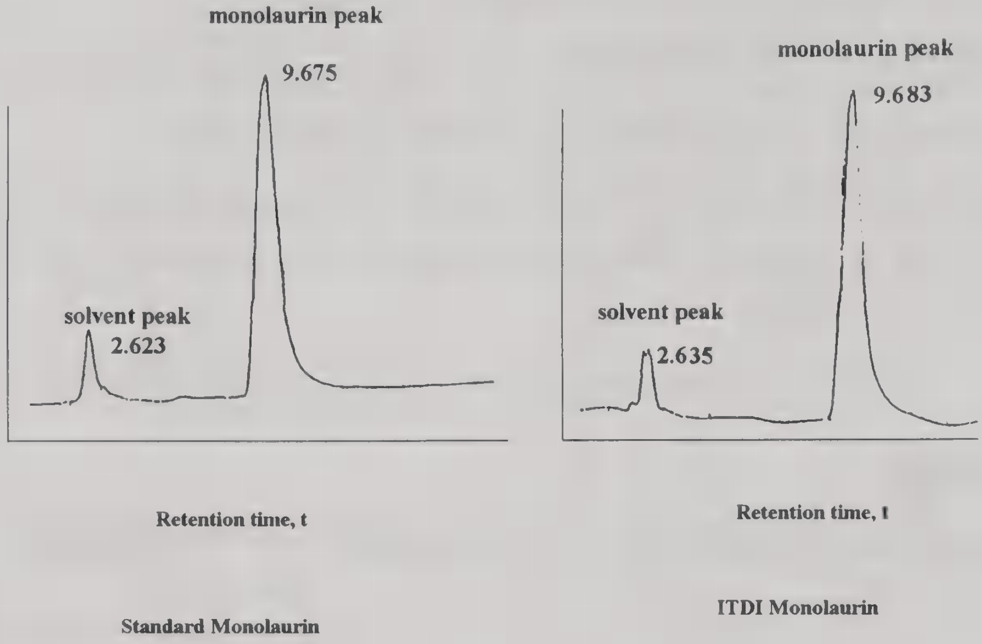


Figure 5. Comparative HPLC chromatogram of the standard monolaurin and ITDI monolaurin.

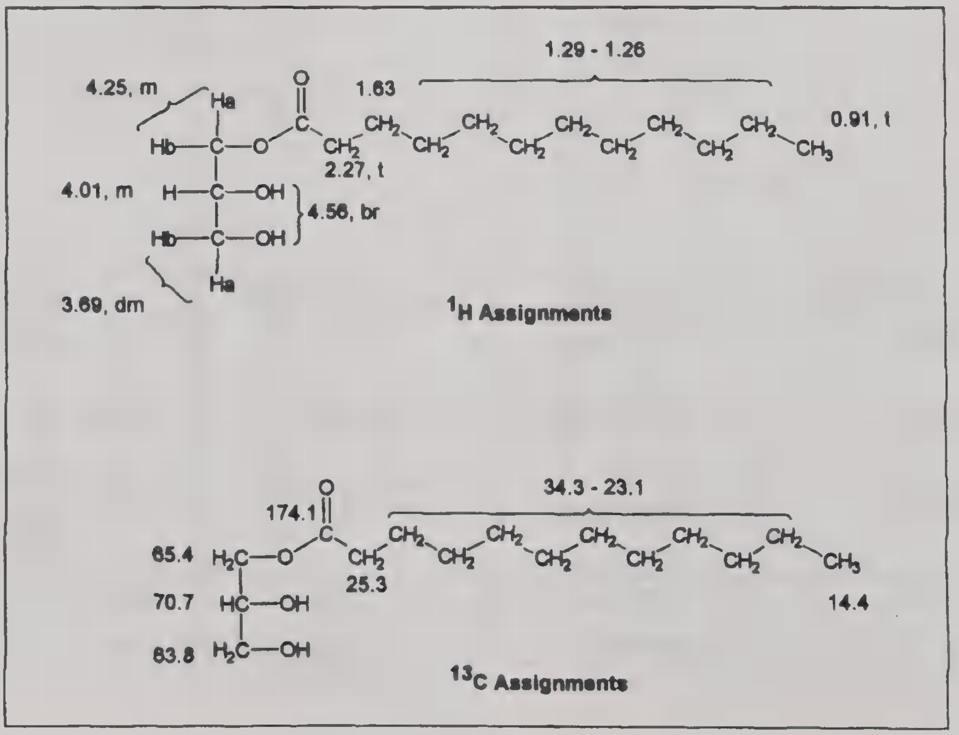
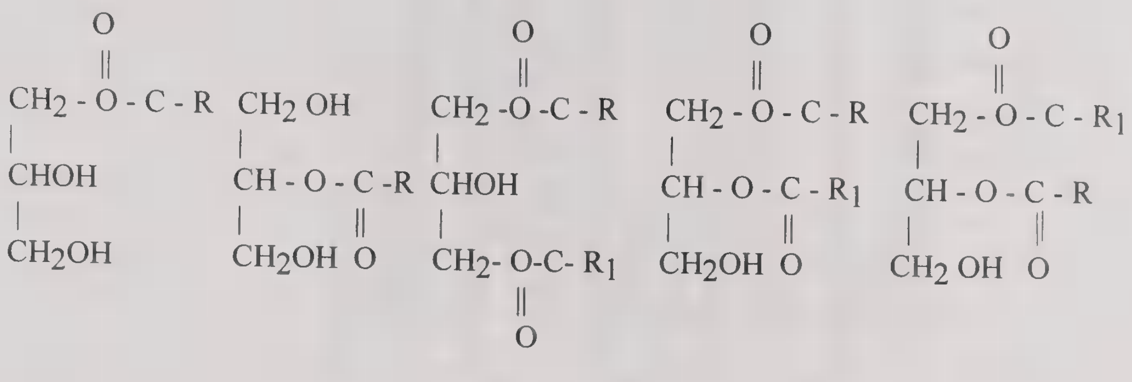


Figure 6. The ^{1}H and ^{13}C assignments resulting from NMR experiments of monolaurin.

RESULTS AND DISCUSSION

Commercial monoglycerides usually contain only 40-60% monoester, 30-45% diester and free glycerol, depending on the ratio of fat to glycerol employed (Swern 1979). Monoglycerides exist in several structural modifications:



1- Mono- 2- Mono- 1,3- Di- 1,2- Di- 1,2- Di-

These conventional monoglycerides are usually used as preservatives, shortenings and emulsifier. With the newly found and a very potential lauric acid as an anti-microbial property, these commercial monoglycerides purity must be improved to meet the specification for food and medicine uses. To develop a high quality monoglyceride rom lauric acid or monolaurin, parameter such as reaction temperature, reaction time and molar ratio were varied and studied their effects on the product. The first parameter to be discussed is the reaction time and its effect on its formation.

Monolaurin formation was favored as time of reaction progressed up to 7th hour, as shown in **Figure 7**. However, while an insignificant increase in monolaurin was observed from the 5th to 7th hour, the di- and trilaurin, on the other hand, were progressively increasing during this period of reaction.

The effect of temperature on the formation of monolaurin is shown in **Figure 8**. While it is evident that monolaurin content increased with reaction temperature from 160 °C to 180 °C, it was also observed that the trilaurin content simultaneously increased as the temperature reaches 180 °C. Beyond this temperature, it was observed that the reaction favored the formation of trilaurin even if glycerol is present in excess.

Development of the nature of esterification catalysts to be used has been directed towards those that are easily removed in the purification process. Among the non-corrosive metallic catalysts used are CaO, nickel, and zinc.

Esterification done in the absence and presence of catalyst was compared The presence of catalyst made the product darker in color. Monolaurin formation attained optimal yield in 5 hours of reaction, as shown in **Figure 9**.

Another reaction parameter, which was investigated, is the molar ratio of the reactants. In order to obtain a product consisting mainly of monoglycerides, glycerol should be present in excess (Viernes *et al* 1990). Although several molar ratios were tried, 1:2.5 and 1:4.0 gave the most promising results. A 1:4.0 molar ratio gave almost the same amount of monolaurin product after 5 h of reaction as shown in **Figure 10**. Increasing the

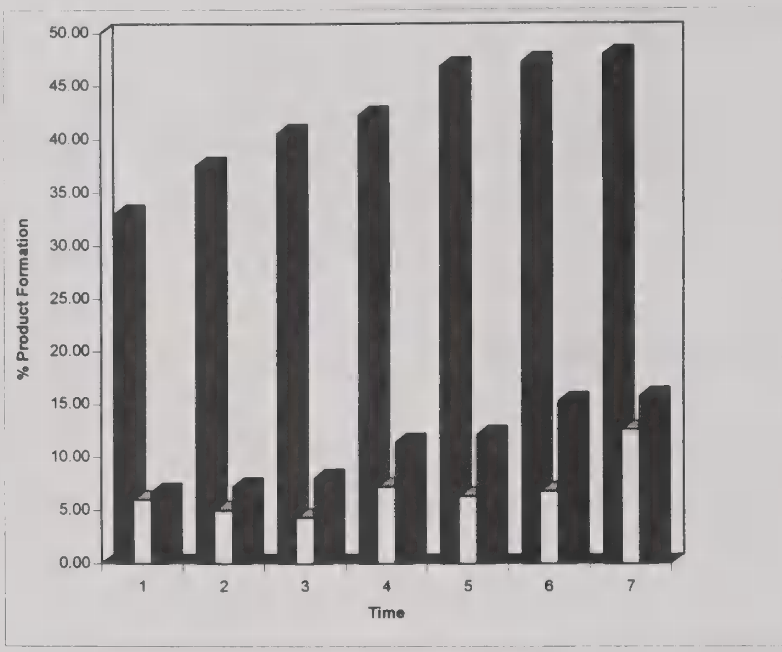


Figure 7. Effect of reaction time on the formation of mono-, di-, and trilaurins (molar ratio = 1:2.5, no catalyst, temperature = 180 °C).

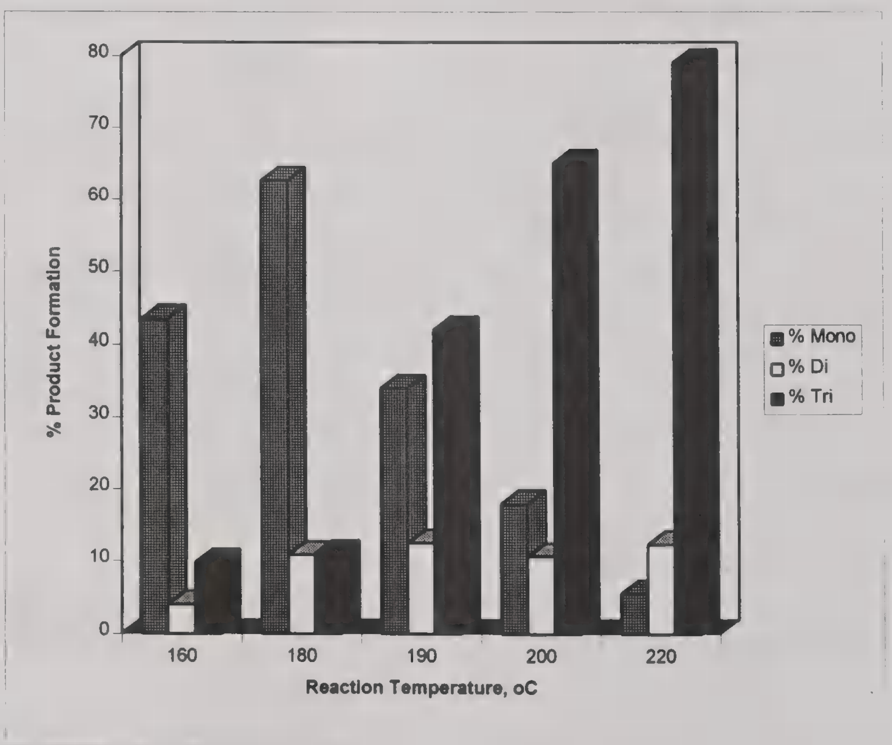


Figure 8. Effect of temperature on the formation of mono-, di- and trilaurins (reaction time = 5 h; molar ratio = 1:4; no catalyst).

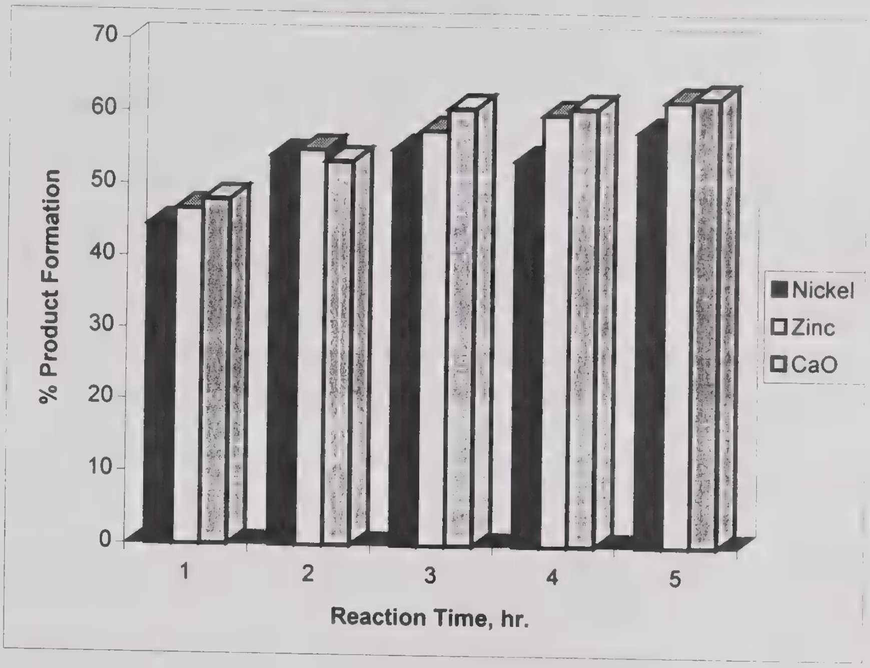


Figure 9. Effect of catalysts on the formation of mono-, di-, and trilaurins (reaction temp. = 180 °C; molar ratio = 1:2.5).

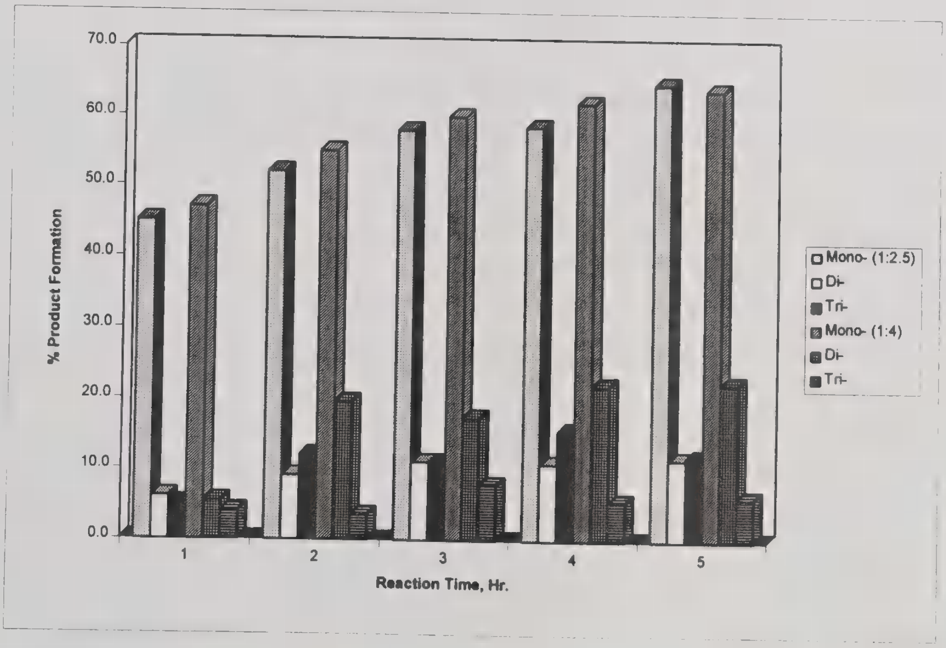


Figure 10. Effect of varying the molar ratio of reactants in the formation of mono-, di-, and trilaurins (reaction temp = 180 °C; no catalyst).

glycerol content by 60% did not increase the monolaurin production significantly. For the economic reason, 1:2.5 molar ratio was used in this study.

CONCLUSIONS

A process has been developed for the preparation of a low volume but high value monolaurin from coconut C_{12} fatty acid. It was established that the following reaction conditions would be best to produce monolaurin in good yield and quality: 55.2% yield; 98.50% purity; reaction time: 5 h; reaction temperature: 180 °C; molar ratio: FA/Glycerol 1:2.5. There were three developed purification methods for crude monolaurin namely, low temperature crystallization method, water refining method and molecular distillation method. Among the three, the latter gave the highest purity and monolaurin.

ACKNOWLEDGEMENTS

Our thanks to Dr. Fabian Dayrit for providing the NMR results and interpretation. Also, we thank to Ms. Merle Villanueva, our Chief, Ms . Ernanda Suavillo, thanks for her time and efforts on the graphs and figures presented in the text, and to the rest of the staff of the Organic Chemical Section of the Chemicals and Mineral Division, who in one way or the other, have helped in the completion of this work.

We would also like to thank Cocochem Philippines for providing the C_{12} fatty acid used throughout the study.

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Effect of Acid Treatment of Fuzzy Cottonseed on Free Fatty Acid Content Determination

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CONTENT

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ABSTRACT

Free fatty acid (FFA) content is one of the many important seed quality indicators. It affects the seed grade and the seed price. Therefore, both accuracy and precision of the FFA method are critical. The determination of free fatty acid (FFA) content in cottonseed is a two step procedure. The old AOCS Official Method Aa 6-38 required a room temperature extraction with petroleum ether followed by desolventization and titration. This method is reproducible but underestimates the FFA of cottonseed by about 10%. To correct the accuracy problem of AOCS Official Method Aa 6-38, a 4-h Butt-type or Soxhlet extraction was recommended based on a collaborative study conducted in 1998. In the AOCS Methods Manual, there is a separate method, Aa 4-38, for the determination of the oil content in fuzzy cottonseed for the seed grading purpose. This method calls for a hydrochloric acid fuming treatment of the short linters of fuzzy cottonseed before grinding then followed by a 4-h Butt-type extraction. To permit the use of the oil obtained from Aa 4-38 for FFA determination by titration will save time and analytical cost. But the effect of the acid treatment of fuzzy cottonseed on its FFA needed to be assessed before the recommendation could be made. This was the purpose of the second collaborative study. The results of this collaborative study presented are: moisture determination by Aa 3-38, oil content by Aa 4-38, FFA in the oil from Aa 4-38 by titration, and FFA in oil from 4-h Butt-type extraction of the mechanically dehulled cottonseed kernels. These assays were done in duplicate for eight different batches of cottonseed samples with various levels of FFA. Limited statistical analysis of these data are presented. The results demonstrated that the effect of acid treatment of fuzzy cottonseed, as described in the AOCS Official Method Aa 4-38, on FFA in the oils of cottonseed is small.

SUMMARY REPORT

Eight cottonseed samples were collected, thoroughly mixed and mailed to each of the 10 labs via Federal Express overnight service by Mr. D.W. Bell, Chickasha Oil Mill, Georgia. (See Appendix for detailed information)

Out of 10 labs, only six labs turned in their results (TABLE 1).

To do an exhaustive (ISO) statistical analysis, minimum 8 labs and 5 samples are required.

Moistures on whole cottonseed samples are very variable and mean values are very unreliable because of missing and high/low values (TABLE 2 & 3).

Moisture determination is more reliable in dehulled kernels.

Oil content values from whole cottonseed samples will be affected by the moisture variability therefore, the seed grade (TABLE 4).

Oil extraction from kernels has <5% variation.

FFA Precision increases with FFA content - see standard deviation (SD) values in the tables (TABLE 5).

FFA in oil from dehulled kernels is close to the values from acid treated whole cottonseed samples.

Results from 8-h exhaustive extraction (AOCS Method Am 2-93) fit well with conventional data - probably fall within normal data population.

From these data it appears that fuming has minimum effect on oil or FFA as shown in FIGURE 1.

COLLABORATIVE STUDY TO EVALUATE THE EFFECT OF ACID TREATMENT ON FREE FATTY ACID (FFA) DETERMINATION IN CS

(December 1999 to January 2000)

TABLE 1 Participating Labs and The Grinder and Grinding Condition Used in Each of the Six Labs

Company 1: A&L Plains Agricultural Labs
- Grinder Make: Micro-sample Mill for grinding fumed seed and dehulled meats
- Grinder Model: not available
- Grinder Setting: Bauer Mill #148 adjusted to just break seed for separating meats from hulls (using sieve/4-mesh screen)
- Screen Size: Herringbone screen size .027
- Observations and Comments:

Company 2: Mid-Continent Labs, Greenwood
- Grinder Make: Mikro
- Grinder Model:
- Grinder Setting:
- Screen Size: 1/8" rd
- Observations and Comments:

Company 3: Mid-Continent Labs, Jackson
- Grinder Make: Mikro
- Grinder Model: Batam
- Grinder Setting:
- Screen Size: .125 rd
- Observations and Comments:

Company 4: Mid-Continent Labs, Memphis
- Grinder Make:Mikro
- Grinder Model: Batam
- Grinder Setting:
- Screen Size: 1/8" rd
- Observations and Comments:

Company 5: Southern Cotton Oil Co.
- Grinder Make:Bantam Micro-sample mill
- Grinder Model:
- Grinder Setting:
- Screen Size: .125 round
- Observations and Comments: Seeds were dehulled using a Bauer Mill and meats were ground through the Bantam Micro Samplemill

Company 6: Woodson-Tenent Labs
- Grinder Make:PUCO
- Grinder Model: 579A
- Grinder Setting:
- Screen Size: .035
- Observations and Comments: Infrared Oven

COLLAB[illegible]IVE STUDY TO EVALUATE THE EFFECT OF ACID TREATMENT ON FRE[illegible]TTY ACID (FFA) DETERMINATION IN COTTONSEED
(December [illegible]9 to January 2000)

TABLE 2 Moisture Content (by AOCS Aa 3-38), Percent Oil (by AOCS Aa 4-38) and FFA in the Extracted Oil from Fresh White Cottonseed

Table 2a Percent Moisture Determination on Fresh Sample using Procedure Aa 3-38

Company	Sample 1			Sample 2			Sample 3			Sample 4			Sample 5			Sample 6			Sample 7			Sample 8		
	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean
1	9.48	9.44	9.46	9.50	9.55	9.53	9.80	9.83	9.82	10.35	10.19	10.27	8.82	8.87	8.85	10.70	10.55	10.63	10.13	10.07	10.10	10.13	9.94	10.04
2	3.76	3.80	3.78	3.86	3.88	3.87	3.70	3.76	3.73	4.06	4.02	4.04	3.46	3.42	3.44	3.08	3.10	3.09	3.34	3.40	3.37	3.46	3.36	3.41
3	2.48	2.54	2.51	1.70	1.76	1.73	1.84	1.96	1.90	2.60	2.50	2.55	2.08	2.04	2.06	1.70	1.94	1.82	1.62	1.56	1.59	2.92	3.00	2.96
4	1.64	1.70	1.67	1.20	1.14	1.17	1.98	2.00	1.99	1.42	1.40	1.41	0.86	0.96	0.91	1.14	1.20	1.17	1.04	1.10	1.07	1.44	1.50	1.47
5	9.70		9.70	10.20		10.20	10.20		10.20	10.50		10.50	8.80		8.80	10.60		10.60	10.00		10.00	10.10		10.10
6	3.75	3.80	3.78	3.88	3.88	3.88	3.24	3.26	3.25	4.27	4.30	4.29	2.48	2.50	2.49	3.34	3.35	3.35	3.15	3.10	3.13	4.21	4.22	4.22
mean	5.14	4.26	5.15	5.06	4.04	5.06	5.13	4.16	5.15	5.53	4.48	5.51	4.42	3.56	4.42	5.09	4.03	5.11	4.88	3.85	4.88	5.38	4.40	5.37
SD	3.23	2.71	3.22	3.54	2.97	3.54	3.51	2.92	3.50	3.59	3.04	3.58	3.20	2.77	3.20	4.00	3.35	3.96	3.75	3.23	3.75	3.45	2.90	3.42

Table 2b Percent Oil Determination on Fresh Sample using Procedure Aa 4-38

Company	Sample 1			Sample 2			Sample 3			Sample 4			Sample 5			Sample 6			Sample 7			Sample 8		
	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean
1	17.92	18.02	17.97	17.30	17.28	17.29	17.76	17.96	17.86	20.62	20.00	20.31	15.26	15.74	15.50	17.06	17.12	17.09	18.02	18.02	18.02	19.28	19.44	19.36
2	18.80	18.70	18.75	18.20	18.30	18.25	18.80	18.80	18.80	21.20	21.10	21.15	17.10	17.20	17.15	17.90	18.00	17.95	19.10	19.10	19.10	20.30	20.10	20.20
3	19.00	18.90	18.95	18.80	18.60	18.70	18.70	18.70	18.70	20.90	21.00	20.95	17.30	17.50	17.40	18.00	17.70	17.85	19.20	19.00	19.10	20.40	20.30	20.35
4	19.10	19.18	19.14	19.11	18.65	18.88	18.60	18.60	18.60	21.12	21.14	21.13	17.44	17.63	17.54	17.96	17.38	17.67	19.23	18.97	19.10	20.28	20.43	20.36
5	17.00	16.80	16.90	16.80	16.60	16.70	17.20	16.90	17.05	18.90	18.70	18.80	16.10	15.90	16.00	16.30	16.00	16.15	17.40	17.40	17.40	18.90	18.70	18.80
6	18.17	18.20	18.19	17.70	17.73	17.72	18.30	18.27	18.29	20.38	20.30	20.34	17.40	17.35	17.38	17.31	17.21	17.26	18.69	18.70	18.70	19.80	19.86	19.83
mean	18.33	18.30	18.32	17.99	17.86	17.92	18.23	18.21	18.22	20.52	20.37	20.45	16.77	16.89	16.83	17.42	17.24	17.33	18.61	18.53	18.57	19.83	19.81	19.82
SD	0.73	0.78	0.75	0.81	0.74	0.77	0.57	0.65	0.61	0.78	0.86	0.81	0.81	0.77	0.78	0.61	0.63	0.61	0.68	0.62	0.65	0.57	0.59	0.57

Table 2c Percent FFA Determination on Fresh Sample using Oil from Procedure Aa 4-38

Company	Sample 1			Sample 2			Sample 3			Sample 4			Sample 5			Sample 6			Sample 7			Sample 8		
	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean
1	6.50	6.40	6.45	1.10	1.10	1.10	8.90	8.80	8.85	11.80	11.30	11.55	1.70	1.60	1.65	17.50	17.40	17.45	6.90	6.80	6.85	10.00	10.20	10.10
2	6.30	6.10	6.20	1.40	1.50	1.45	10.20	10.30	10.25	12.00	12.00	12.00	1.50	1.40	1.45	16.50	16.70	16.60	6.60	6.40	6.50	8.30	8.20	8.25
3	6.00	6.10	6.05	0.90	0.90	0.90	8.50	8.60	8.55	11.20	11.40	11.30	1.40	1.50	1.45	15.60	15.70	15.65	5.00	5.20	5.10	8.20	8.20	8.20
4	6.00	5.80	5.90	1.70	1.20	1.45	8.30	8.40	8.35	10.80	10.70	10.75	1.50	1.60	1.55	16.60	16.60	16.60	4.70	5.10	4.90	8.10	7.70	7.90
5	8.40	8.40	8.40	1.20	1.00	1.10	9.40	9.70	9.55	11.20	11.00	11.10	1.60	1.60	1.60	17.20	17.00	17.10	7.40	7.10	7.25	8.90	9.10	9.00
6	7.76	7.80	7.78	1.59	1.59	1.59	10.58	10.60	10.59	12.44	12.40	12.42	4.87	4.88	4.88	16.35	16.40	16.38	9.00	9.10	9.05	10.59	10.55	10.57
mean	6.83	6.77	6.80	1.32	1.22	1.27	9.31	9.40	9.36	11.57	11.47	11.52	2.10	2.10	2.10	16.63	16.63	16.63	6.60	6.62	6.61	9.02	8.99	9.00
SD	0.92	0.97	0.95	0.28	0.25	0.25	0.84	0.85	0.84	0.56	0.58	0.56	1.24	1.25	1.25	0.61	0.52	0.57	1.45	1.34	1.39	0.96	1.07	1.01

COLLABORATIVE STUDY TO EVALUATE THE EFFECT OF ACID TREATMENT ON FREE FATTY ACID (FFA) DETERMINATION IN CS

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TABLE 3 Moisture Content (by AOCS Ba 2-38), Percent Oil (by 4-h Butt-type Extraction or AOCS Am2-93) and FFA in the Extracted Oil from Mechanically Dehulled Cottonseed Kernels

Table 3a Percent Moisture Determination of Mechanically Dehulled Kernels using Procedure Ba 2-38

Company	Sample 1			Sample 2			Sample 3			Sample 4			Sample 5			Sample 6			Sample 7			Sample 8		
	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean
1	8.24	8.20	8.22	7.82	7.92	7.87	8.46	8.76	8.61	8.90	8.80	8.85	7.52	7.28	7.40	8.84	8.72	8.78	8.36	8.30	8.33	8.10	8.18	8.14
2	8.36	8.38	8.37	8.36	8.32	8.34	8.60	8.64	8.62	9.01	9.10	9.06	7.71	7.82	7.77	9.01	9.05	9.03	8.68	8.62	8.65	8.41	8.44	8.43
3	8.76	8.74	8.75	8.44	8.48	8.46	8.68	8.86	8.77	9.12	8.84	8.98	7.80	7.78	7.79	9.28	9.22	9.25	8.80	8.86	8.83	8.64	8.60	8.62
4	8.66	8.60	8.63	8.48	8.40	8.44	8.96	8.90	8.93	9.04	9.88	9.46	7.92	7.86	7.89	9.28	9.20	9.24	8.28	8.60	8.44	8.68	8.60	8.64
5	8.30		8.30	8.30		8.30	8.60		8.60	8.70		8.70	7.50		7.50	8.70		8.70	8.50		8.50	8.50		8.50
6	8.22	8.26	8.24	7.97	8.00	7.99	8.51	8.48	8.50	8.90	8.90	8.90	7.60	7.66	7.63	9.19	9.22	9.21	8.53	8.60	8.57	8.38	8.36	8.37
mean	8.42	8.44	8.42	8.23	8.22	8.23	8.64	8.73	8.67	8.95	9.10	8.99	7.68	7.68	7.66	9.05	9.08	9.03	8.53	8.60	8.55	8.45	8.44	8.45
SD	0.21	0.20	0.20	0.25	0.22	0.22	0.16	0.15	0.14	0.13	0.40	0.24	0.15	0.21	0.17	0.22	0.19	0.22	0.18	0.18	0.16	0.19	0.16	0.17

Tabke 3b Percent Oil Determination of Mechanically Dehulled Kernels using 4-h Butt-type Extraction

Company	Sample 1			Sample 2			Sample 3			Sample 4			Sample 5			Sample 6			Sample 7			Sample 8		
	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean
1	31.68	31.76	31.72	33.10	33.22	33.16	34.36	34.70	34.53	34.16	34.38	34.27	29.90	29.90	29.90	31.50	31.44	31.47	32.44	32.46	32.45	31.62	32.04	31.83
2	30.00	29.80	29.90	30.70	30.20	30.45	32.60	32.50	32.55	32.50	32.50	32.50	27.20	27.00	27.10	29.80	29.90	29.85	30.40	30.50	30.45	31.80	31.90	31.85
3	29.20	29.00	29.10	30.00	30.20	30.10	31.10	30.00	30.55	31.10	31.20	31.15	26.00	25.90	25.95	28.70	28.80	28.75	29.00	29.10	29.05	30.50	30.30	30.40
4	28.19	28.77	28.48	30.07	29.88	29.98	31.35	30.96	31.16	31.33	31.70	31.52	26.22	26.03	26.13	28.87	28.62	28.75	29.13	29.28	29.21	30.70	30.38	30.54
5	31.00	30.80	30.90	31.70	31.30	31.50	33.20	32.90	33.05	33.20	33.10	33.15	28.30	28.10	28.20	30.30	30.50	30.40	31.30	31.10	31.20	33.10	32.90	33.00
6	30.86	30.90	30.88	30.42	30.42	30.42	31.23	31.08	31.16	33.11	33.20	33.16	25.92	25.72	25.82	29.16	29.06	29.11	29.79	30.00	29.90	31.11	31.20	31.16
mean	30.16	30.17	30.16	31.00	30.87	30.93	32.31	32.02	32.17	32.57	32.68	32.62	27.26	27.11	27.18	29.72	29.72	29.72	30.34	30.41	30.38	31.47	31.45	31.46
SD	1.18	1.07	1.12	1.09	1.14	1.11	1.20	1.54	1.36	1.07	1.04	1.06	1.44	1.49	1.47	0.97	1.01	0.98	1.22	1.14	1.18	0.86	0.93	0.89

Table 3c Percent FFA Determination of Mechanically Dehulled Kernels using oil from 4-h Butt-type Extraction

Company	Sample 1			Sample 2			Sample 3			Sample 4			Sample 5			Sample 6			Sample 7			Sample 8		
	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean
1	7.10	7.20	7.15	0.70	0.70	0.70	9.90	9.80	9.85	11.80	12.10	11.95	1.70	1.70	1.70	18.50	18.70	18.60	6.00	6.20	6.10	8.80	8.80	8.80
2	5.80	5.70	5.75	0.90	0.90	0.90	7.80	7.70	7.75	11.60	11.40	11.50	1.30	1.40	1.35	16.10	16.20	16.15	6.90	6.80	6.85	8.80	8.80	8.80
3	6.50	6.60	6.55	1.10	1.10	1.10	8.00	8.00	8.00	10.70	11.00	10.85	1.90	1.80	1.85	17.90	17.80	17.85	5.00	5.20	5.10	8.20	8.20	8.20
4	7.20	7.40	7.30	1.30	0.90	1.10	8.80	8.70	8.75	12.20	12.10	12.15	1.50	1.60	1.55	17.60	17.10	17.35	5.20	5.20	5.20	8.70	8.40	8.55
5	6.90	6.90	6.90	0.80	1.10	0.95	9.70	9.80	9.75	10.60	11.10	10.85	1.40	1.60	1.50	16.60	16.60	16.60	6.50	6.70	6.60	9.20	9.50	9.35
6	6.43	6.45	6.44	1.37	1.37	1.37	11.75	11.68	11.72	14.80	14.77	14.79	3.34	3.30	3.32	17.35	17.45	17.40	8.30	8.42	8.36	10.26	10.35	10.31
mean	6.66	6.71	6.68	1.03	1.01	1.02	9.33	9.28	9.30	11.95	12.08	12.01	1.86	1.90	1.88	17.34	17.31	17.33	6.32	6.42	6.37	8.99	9.01	9.00
SD	0.48	0.56	0.52	0.25	0.21	0.21	1.34	1.34	1.34	1.40	1.28	1.33	0.69	0.64	0.66	0.80	0.81	0.80	1.11	1.10	1.10	0.64	0.72	0.68

Table 3d Percent Oil Determination of Mechanically Dehulled Kernels using Procedure Am 2-93

Company	Sample 1			Sample 2			Sample 3			Sample 4			Sample 5			Sample 6			Sample 7			Sample 8		
	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean
3	28.10	28.32	28.21	30.10	30.20	30.15	31.20	31.30	31.25	31.30	31.40	31.35	26.90	27.00	26.95	29.00	29.10	29.05	29.30	29.20	29.25	30.60	30.50	30.55
4	28.29	28.45	28.37	30.00	30.44	30.22	31.36	31.50	31.43	31.54	31.30	31.42	27.04	26.88	26.96	29.11	28.94	29.03	29.45	29.35	29.40	30.75	30.85	30.80
mean	28.20	28.39	28.29	30.05	30.32	30.19	31.28	31.40	31.34	31.42	31.35	31.39	26.97	26.94	26.96	29.06	29.02	29.04	29.38	29.28	29.33	30.68	30.68	30.68

Table 3e Percent FFA Determination of Mechanically Dehulled Kernels using Oil from Procedure Am 2-93

Company	Sample 1			Sample 2			Sample 3			Sample 4			Sample 5			Sample 6			Sample 7			Sample 8		
	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean	Rep #1	Rep #2	mean
3	6.40	6.60	6.50	1.20	1.30	1.25	8.60	8.40	8.50	11.90	11.80	11.85	1.30	1.50	1.40	17.20	17.40	17.30	4.90	5.30	5.10	8.30	8.20	8.25
4	7.00	7.30	7.15	1.40	1.20	1.30	8.60	8.40	8.50	11.80	12.00	11.90	1.30	1.60	1.45	17.10	17.60	17.35	4.90	5.30	5.10	8.30	8.10	8.20
mean	6.70	6.95	6.83	1.30	1.25	1.28	8.60	8.40	8.50	11.85	11.90	11.88	1.30	1.55	1.43	17.15	17.50	17.33	4.90	5.30	5.10	8.30	8.15	8.23

COLLABORATIVE STUDY TO EVALUATE THE EFFECT OF ACID TREATMENT ON FREE FATTY ACID (FFA) DETERMINATION IN CS

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TABLE 4 Summary Data Sheet of Mean Values and Standard Deviations

Table 4a Percent Moisture Determination on Fresh Sample using Procedure Aa 3-38

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
mean	5.15	5.06	5.15	5.51	4.42	5.11	4.88	5.37
SD	3.22	3.54	3.50	3.58	3.20	3.96	3.75	3.42

Table 4b Percent Oil Determination on Fresh Sample using Procedure Aa 4-38

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
mean	18.32	17.92	18.22	20.45	16.83	17.33	18.57	19.82
SD	0.75	0.77	0.61	0.81	0.78	0.61	0.65	0.57

Table 4c Percent FFA Determination on Fresh Sample using Oil from Procedure Aa 4-38

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
mean	6.80	1.27	9.36	11.52	2.10	16.63	6.61	9.00
SD	0.95	0.25	0.84	0.56	1.25	0.57	1.39	1.01

Table 4d Percent Moisture Determination of Mechanically Dehulled Kernels using Procedure Ba 2-38

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
mean	8.42	8.23	8.67	8.99	7.66	9.03	8.55	8.45
SD	0.20	0.22	0.14	0.24	0.17	0.22	0.16	0.17

Table 4e Percent Oil Determination of Mechanically Dehulled Kernels using 4-h Butt-type Extraction

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
mean	30.16	30.93	32.17	32.62	27.18	29.72	30.38	31.46
SD	1.12	1.11	1.36	1.06	1.47	0.98	1.18	0.89

Table 4f Percent FFA Determination of Mechanically Dehulled Kernels using oil from 4-h Butt-type Extraction

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
mean	6.68	1.02	9.30	12.01	1.88	17.33	6.37	9.00
SD	0.52	0.21	1.34	1.33	0.66	0.80	1.10	0.68

Table 4g Percent Oil Determination of Mechanically Dehulled Kernels using Procedure Am 2-93

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
mean	28.29	30.19	31.34	31.39	26.96	29.04	29.33	30.68

Table 4h Percent FFA Determination of Mechanically Dehulled Kernels using Oil from Procedure Am 2-93

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
mean	6.83	1.28	8.50	11.88	1.43	17.33	5.10	8.23

COLLABORATIVE STUDY TO EVALUATE THE EFFECT OF ACID TREATMENT ON FREE FATTY ACID (FFA) DETERMINATION IN CS
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TABLE 5 FFA Summary Sheet

Table 5a Percent FFA Determination on Fresh Sample using Oil from Procedure Aa 4-38

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
mean	6.80	1.27	9.36	11.52	2.10	16.63	6.61	9.00
SD	0.95	0.25	0.84	0.56	1.25	0.57	1.39	1.01

Table 5b Percent FFA Determination of Mechanically Dehulled Kernels using oil from 4-h Butt-type Extraction

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
mean	6.68	1.02	9.30	12.01	1.88	17.33	6.37	9.00
SD	0.52	0.21	1.34	1.33	0.66	0.80	1.10	0.68

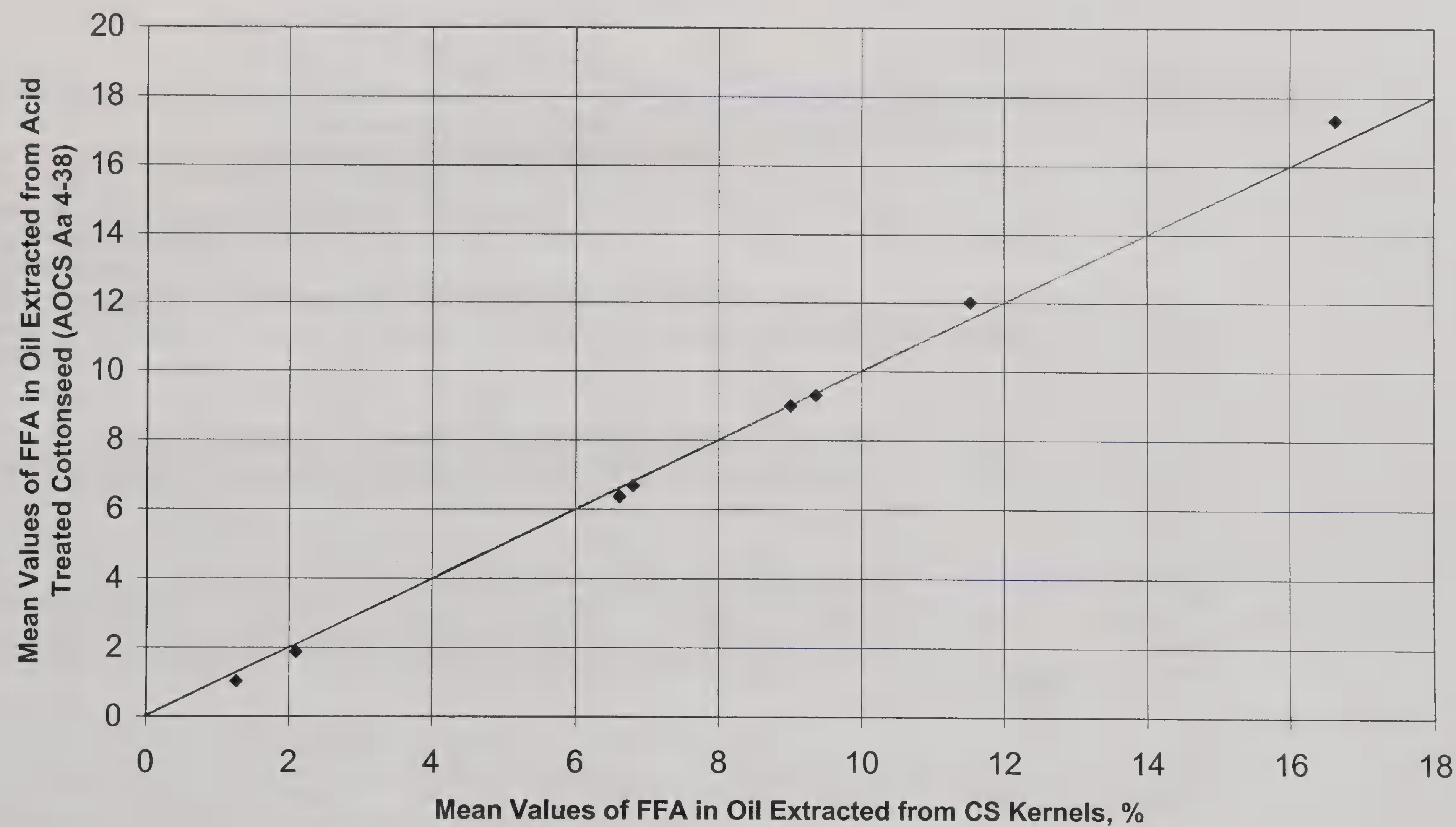
Table 5c Percent FFA Determination of Mechanically Dehulled Kernels using Oil from Procedure Am 2-93

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
mean	6.83	1.28	8.50	11.88	1.43	17.33	5.10	8.23

Comments:

1. There are too few labs to do an exhaustive (ISO) statistical analysis. We need 8 or 9 (so we can remove 2/9 as outliers)
2. Moisture on fresh samples very variable - mean values are very unreliable because of missing and high/low values
3. Oil values from fresh samples will be affected by the moisture variability
4. FFA precision increases with FFA content - see SD values
5. Moisture determination more reliable in dehulled kernels
6. Oil extraction from kernels has <5% variation
7. FFA in oil from dehulled kernels is close to the values from fresh samples
8. Results from Am 2-93 fit well with conventional data - probably fall within normal data population
9. From these data it appears that fuming has minimum effect on oil or FFA, but the whole seed moistures showed large variations.

FIGURE 1. Percent FFA in Oil Extracted from Acid Treated CS (Aa 4-38) versus Percent FFA in Oil Extracted from CS Kernels from Eight Samples and Six Labs



APPENDIX - Experimental Protocol

COLLABORATIVE STUDY TO EVALUATE THE EFFECT OF ACID TREATMENT OF THE WHOLE COTTONSEED ON ITS FREE FATTY ACID DETERMINATION
January 2000
(Revised)

BACKGROUND: The collaborative study conducted in the summer of 1998 demonstrated that AOCS Method Aa 6-38 underestimated the amount of free fatty acid (FFA) in cottonseed by about 10% when compared with a four hour Butt type or Soxhlet extraction. Therefore, a minimum 4 hour Butt type or Soxhlet extraction was recommended to AOCS and the cottonseed industry for the determination of FFA in cottonseed. Since AOCS Method Aa 4-38 uses 4-h Butt type extraction to determine the oil content in acid treated white cottonseed, it will be ideal that this oil can be used for the FFA measurement.

OBJECTIVE: To assess the effect of acid treatment of white cottonseed as described in AOCS Aa 4-38 on the amount of FFA in the oil obtained by this method.

APPROACH:

(1) Eight lots of thoroughly mixed cottonseed samples with a range of FFA, 1 pound each, will be mailed to participating labs by overnight shipment.

(2) Determine oil content of the eight cottonseed samples after acid treatment according to AOCS Aa 4-38 and the moisture content of the acid treated seed according to the procedure described in AOCS Aa 3-38. **Please grind the acid treated seed to less than 1 mm sieve.**

(3) Determine FFA in the oil obtained from (2).

(4) Dehull each batch of cottonseed sample with a blender, such as Waring Blender, and obtain adequate meats for (a) moisture determination in the meats following AOCS Ba 2-38; (b) oil content determination in the meats using 4-h Butt type extract similar to that of Aa 4-38 and please **grind the meats to less than 1 mm sieve** prior to weighing and extraction; and (c) FFA determination by titration with the oil sample obtained from (b).

(5) Repeat (2) and (4) for each lot of cottonseed sample.

(6) Please complete analyzing all the samples within a week from the time the cottonseed samples are received.

(7) Record the data and comment on any unusual observations in the attached table **and please indicate the acid treatment of the fuzzy seed in (2) is done in a Henry Oven or Infrared Oven.**

(8) Analyze all the meat samples obtained from (4) in duplicate with AOCS Method Am 2-93 (FOSFA International Method; copy of the method attached) for oil content and determine the FFA in the oil extracted by AOCS Am 2-93. [This assay is optional. But these data will strengthen the final results of the collaborative study.]

(9) Keep the remaining cottonseed samples in refrigeration temperature until all the data are received and analyzed by the Southern Regional Research Center, Agricultural Research Service, USDA in New Orleans, Louisiana.

(10) Send the results to P. J. Wan at the Southern Regional Research Center, 1100 Robert E. Lee Blvd., New Orleans, LA 70124, or by FAX at 504-286-4419 or by E-mail: pwan@nola.srrc.usda.gov.

EXPECTED RESULTS: You will review the entire set of the data collected along with the statistical analysis of these data and the final proposed recommendation to AOCS for the determination of FFA in cottonseed. We would like to finish this effort by the end of February, 2000.

COLLABORATIVE STUDY TO EVALUATE THE EFFECT OF ACID TREATMENT ON FREE FATTY ACID DETERMINATION IN COTTONSEED

			On Fresh Sample			Mechanically dehulled kernels			Mechanically dehulled kernels			
		Replicate	Aa 3-38	Aa 4-38		Ba 2-38	By 4-h Butt type Extraction		Am 2-93		Type of Grinder	
Date	Sample ID	Number	% moist.	% oil	% FFA	% moist.	% Oil	% FFA	% oil	% FFA	Make/Model/Screen/Settings	Observations & Comments

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January, 2000

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